STIC Search Report
EIC 1700

STIC Database Tracking Number: 134467

TO: Eisa Elhilo
Location: REM 9A60
Art Unit : 1751
October 6, 2004

Case Serial Number: 10/656423

From: Kathleen Fuller
Location: EIC 1700
REMSEN 4B28
Phone: 571/272-2505
Kathleen.Fuller@uspto.gov

Search Notes

There were 57 structures from the structure search. Of the 14 CA references from the 57 structures only one was on the utility and it was the applicant. I printed the other 13 CA references which have no hair or kerat? Utility,.

STIC Search Results Feedback Form

EIC17000

Questions about the scope or the results of the search? Contact *the EIC searcher or contact:*

Kathleen Fuller, EIC 1700 Team Leader
571/272-2505 REMSEN 4B28

Voluntary Results Feedback Form

- *I am an examiner in Workgroup:* ☐ *Example: 1713*
- *Relevant prior art* ***found****, search results used as follows:*
  - ☐ 102 rejection
  - ☐ 103 rejection
  - ☐ Cited as being of interest.
  - ☐ Helped examiner better understand the invention.
  - ☐ Helped examiner better understand the state of the art in their technology.

Types of relevant prior art found:

- ☐ Foreign Patent(s)
- ☐ Non-Patent Literature
  (journal articles, conference proceedings, new product announcements etc.)

- *Relevant prior art* ***not found:***
  - ☐ Results verified the lack of relevant prior art (helped determine patentability).
  - ☐ Results were not useful in determining patentability or understanding the invention.

Comments:

Drop off or send completed forms to EIC1700 REMSEN 4B28

=> FILE REG
FILE 'REGISTRY' ENTERED AT 11:50:45 ON 06 OCT 2004
USE IS SUBJECT TO THE TERMS OF YOUR STN CUSTOMER AGREEMENT.
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Property values tagged with IC are from the ZIC/VINITI data file provided by InfoChem.

STRUCTURE FILE UPDATES: 4 OCT 2004 HIGHEST RN 756793-93-8
DICTIONARY FILE UPDATES: 4 OCT 2004 HIGHEST RN 756793-93-8

TSCA INFORMATION NOW CURRENT THROUGH MAY 21, 2004

Please note that search-term pricing does apply when conducting SmartSELECT searches.

Crossover limits have been increased. See HELP CROSSOVER for details.

Experimental and calculated property data are now available. For more information enter HELP PROP at an arrow prompt in the file or refer to the file summary sheet on the web at:
http://www.cas.org/ONLINE/DBSS/registryss.html

=> FILE HCAPLU
FILE 'HCAPLUS' ENTERED AT 11:50:50 ON 06 OCT 2004
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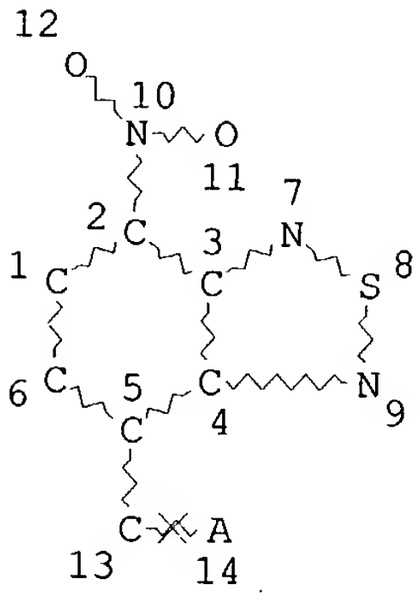
FILE COVERS 1907 - 6 Oct 2004 VOL 141 ISS 15
FILE LAST UPDATED: 5 Oct 2004 (20041005/ED)

This file contains CAS Registry Numbers for easy and accurate substance identification.

=> D QUE
L36 STR

KATHLEEN FULLER EIC 1700 REMSEN 4B28 571/272-2505



← 57 structures from this query

NODE ATTRIBUTES:
NSPEC IS RC AT 13
NSPEC IS RC AT 14
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS 14

STEREO ATTRIBUTES: NONE
L38 57 SEA FILE=REGISTRY SSS FUL L36
L40 14 SEA FILE=HCAPLUS ABB=ON L38
L41 1 SEA FILE=HCAPLUS ABB=ON L40 AND (HAIR OR KERAT?)

14 CA references

Only one on utility

=> D L41 BIB ABS IND HITSTR

L41 ANSWER 1 OF 1 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 2002:733847 HCAPLUS
DN 137:247705
TI Preparation of 7-Nitro-2,1,3-benzoxadiazole and 7-Nitro-2,1,3-benzthiadiazole derivatives as **hair** dyes
IN Umbricht, Gisela; Braun, Hans Juergen; Oberson, Sylviane; Mueller, Catherine
PA Wella AG, Germany
SO Ger. Offen., 16 pp.
CODEN: GWXXBX
DT Patent
LA German
FAN.CNT 1

	PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
PI	DE 10113699	A1	20020926	DE 2001-10113699	20010321
	WO 2002076961	A1	20021003	WO 2001-EP12806	20011106

W: AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM
RW: GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF,

applicant

KATHLEEN FULLER EIC 1700 REMSEN 4B28 571/272-2505

BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG
EP 1261591 A1 20021204 EP 2001-274021 20011106
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR
BR 2001010959 A 20030415 BR 2001-10959 20011106
JP 2004518764 T2 20040624 JP 2002-576221 20011106
US 2003171594 A1 20030911 US 2002-276140 20021112
US 6726730 B2 20040427
US 2004139562 A1 20040722 US 2004-752605 20040107
US 2004139563 A1 20040722 US 2004-752606 20040107
PRAI DE 2001-10113699 A 20010321
WO 2001-EP12806 W 20011106
US 2002-276140 A3 20021112
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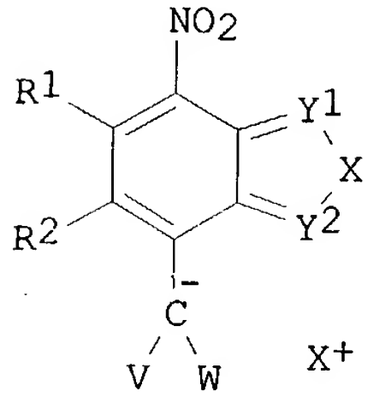
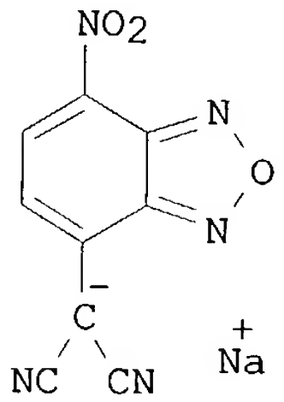
GI



I



II

AB 4-Nitro-2,1,3-benzoxadiazoles and 4-Nitro-2,1,3-benzthiadiazoles I [X = S, O; Y1-2 = N, NO, etc; R1-2 = OH, halo, alkyl, etc.; V = OH, alkyl, aryl, etc.; W = CN, CO, etc.] as coloring agent for **keratin** fibers. For instance, 4-(dicyanomethyl)-7-nitro-2,1,3-benzoxazadiazole sodium salt (II) was prepared from malononitrile, 4-chloro-7-nitro-2,1,3-benzoxazadiazole and sodium carbonate in EtOH in >95% yield. **Hair** was contacted with a solution of 2.5 mmol of II, 5.0 g EtOH, 2.0 g decyl glucoside and 0.2 g Na2EDTA/100 g H2O for 30 min at 40°, rinsed, shampooed, rinsed and dried to show a deep violet color with L = +25.17, a = +54.12 and b = -24.03.

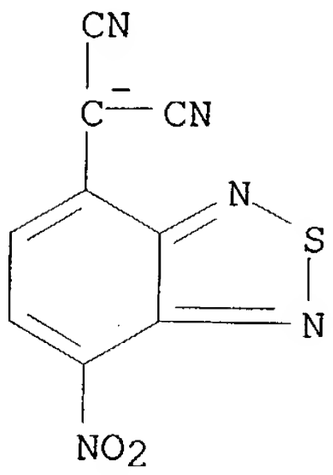
IC ICM C07D271-12
ICS C07D285-14; A61K007-13
CC 28-10 (Heterocyclic Compounds (More Than One Hetero Atom))
Section cross-reference(s): 41, 62
ST benzoxadiazole benzothiadiazole **keratin** fiber prepn
IT **Hair** preparations
(preparation of 7-Nitro-2,1,3-benzoxadiazole and 7-Nitro-2,1,3-benzthiadiazole derivs. as **hair** dyes)
IT Alkali metal salts
RL: BUU (Biological use, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); USES (Uses)
(preparation of 7-Nitro-2,1,3-benzoxadiazole and 7-Nitro-2,1,3-benzthiadiazole derivs. as **hair** dyes)
IT 460722-91-2P, 4-(Dicyanomethyl)-7-nitro-2,1,3-benzoxadiazole sodium salt 460722-92-3P, 4-(1-Cyano-2-ethoxy-2-oxoethyl)-7-nitro-2,1,3-benzoxadiazole sodium salt 460722-93-4P, 4-(Dicyanomethyl)-7-nitro-2,1,3-benzoxadiazol-N-oxide sodium salt **460722-94-5P**, 4-(Dicyanomethyl)-7-nitro-2,1,3-benzthiadiazole sodium salt 460722-95-6P 460722-96-7P, 4-(1-Cyano-3,3-dimethyl-2-oxobutyl)-7-nitro-2,1,3-benzoxadiazole sodium salt 460722-97-8P, 4-(Bis(methoxycarbonyl)methyl)-7-nitro-2,1,3-

benzoxadiazole sodium salt 460722-98-9P 460722-99-0P 460723-00-6P, 4-((Cyano)(carbamoyl)methyl)-7-nitro-2,1,3-benzoxadiazole sodium salt 460723-01-7P **460723-02-8P**, 4-(1-Cyano-2-ethoxy-2-oxoethyl)-7-nitro-2,1,3-benzothiadiazole sodium salt **460723-03-9P** 460723-04-0P 460723-05-1P, 4-(2-Ethoxy-1-nitro-2-oxoethyl)-7-nitro-2,1,3-benzoxadiazole sodium salt 460723-06-2P 460723-07-3P, 4-(1,3-Dioxoindan-2-yl)-7-nitro-2,1,3-benzoxadiazole sodium salt 460723-08-4P, 4-(2-Oxo-2,3-dihydro-1H-indol-3-yl)-7-nitro-2,1,3-benzoxadiazole sodium salt 460723-09-5P, 4-(4-Oxo-2-thioxothiazolidin-5-yl)-7-nitro-2,1,3-benzoxadiazole sodium salt 460723-10-8P 460723-11-9P, 4-(1-Cyano-2-oxo-2-phenylethyl)-2,1,3-benzoxadiazole sodium salt 460723-12-0P 460723-13-1P, 4-(Cyano(4-nitrophenyl)methyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-14-2P** **460723-15-3P**, 4-(1-Cyano-3,3-dimethyl-2-oxobutyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-16-4P**, 4-(Bis(methoxycarbonyl)methyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-17-5P 460723-18-6P 460723-19-7P**, 4-((Carboxy)(cyano)methyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-20-0P**, 4-(2-Ethoxy-1-nitro-2-oxoethyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-21-1P**, 4-[(Aminocarbonyl)cyanomethyl]-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-22-2P 460723-23-3P**, 4-(1,3-Dioxoindan-2-yl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-24-4P**, 4-(2-Oxo-2,3-dihydro-1H-indol-3-yl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-25-5P**, 4-(4-Oxo-2-thioxothiazolidin-5-yl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-26-6P** 460723-27-7P, 4-(1-Cyano-2-oxo-2-phenylethyl)-2,1,3-benzothiadiazole sodium salt
RL: BUU (Biological use, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); USES (Uses)
(preparation of 7-Nitro-2,1,3-benzoxadiazole and 7-Nitro-2,1,3-benzthiadiazole derivs. as **hair** dyes)

IT 67-52-7, Barbituric acid 105-56-6, Ethyl cyanoacetate 107-91-5, 2-Cyanoacetamide 108-59-8, Dimethylmalonate 109-77-3, Malononitrile 555-21-5, 4-Nitrophenylacetonitrile 3524-07-0 6583-06-8, 4-Nitro-2,1,3-benzothiadiazole 10199-89-0, 4-Chloro-7-nitro-2,1,3-benzoxadiazole 18378-13-7 19735-89-8, 1-Phenyl-3-methylpyrazol-5-one 59997-51-2, Pivaloylacetonitrile
RL: RCT (Reactant); RACT (Reactant or reagent)
(reactant; preparation of 7-Nitro-2,1,3-benzoxadiazole and 7-Nitro-2,1,3-benzthiadiazole derivs. as **hair** dyes)

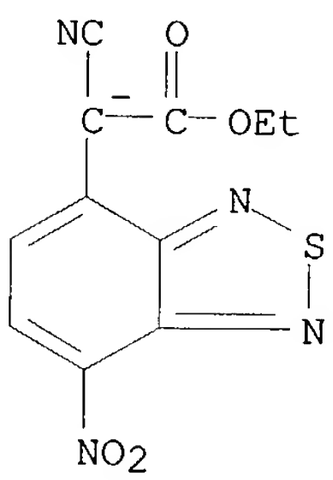
IT **460722-94-5P**, 4-(Dicyanomethyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-02-8P**, 4-(1-Cyano-2-ethoxy-2-oxoethyl)-7-nitro-2,1,3-benzothiadiazole sodium salt **460723-03-9P** **460723-14-2P 460723-15-3P**, 4-(1-Cyano-3,3-dimethyl-2-oxobutyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-16-4P**, 4-(Bis(methoxycarbonyl)methyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-17-5P 460723-18-6P 460723-19-7P**, 4-((Carboxy)(cyano)methyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-20-0P**, 4-(2-Ethoxy-1-nitro-2-oxoethyl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-21-1P**, 4-[(Aminocarbonyl)cyanomethyl]-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-22-2P 460723-23-3P**, 4-(1,3-Dioxoindan-2-yl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-24-4P**, 4-(2-Oxo-2,3-dihydro-1H-indol-3-yl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-25-5P**, 4-(4-Oxo-2-thioxothiazolidin-5-yl)-7-nitro-2,1,3-benzthiadiazole sodium salt **460723-26-6P**
RL: BUU (Biological use, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); USES (Uses)
(preparation of 7-Nitro-2,1,3-benzoxadiazole and 7-Nitro-2,1,3-

benzthiadiazole derivs. as **hair** dyes)
RN 460722-94-5 HCAPLUS
CN Propanedinitrile, (7-nitro-2,1,3-benzothiadiazol-4-yl)-, ion(1-), sodium (9CI) (CA INDEX NAME)



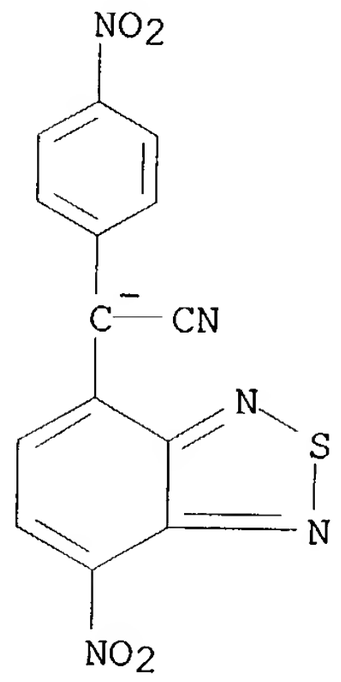
● Na^+

RN 460723-02-8 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-acetic acid, α-cyano-7-nitro-, ethyl ester, ion(1-), sodium (9CI) (CA INDEX NAME)



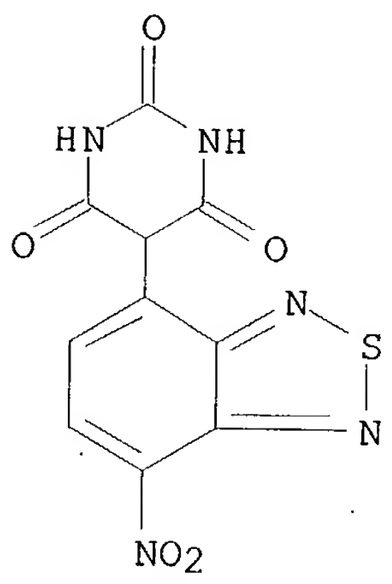
● Na^+

RN 460723-03-9 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-acetonitrile, 7-nitro-α-(4-nitrophenyl)-, ion(1-), sodium (9CI) (CA INDEX NAME)



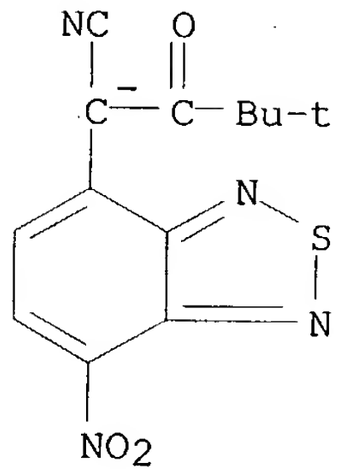
● Na^+

RN 460723-14-2 HCAPLUS
CN 2,4,6(1H,3H,5H)-Pyrimidinetrione, 5-(7-nitro-2,1,3-benzothiadiazol-4-yl)-, monosodium salt (9CI) (CA INDEX NAME)



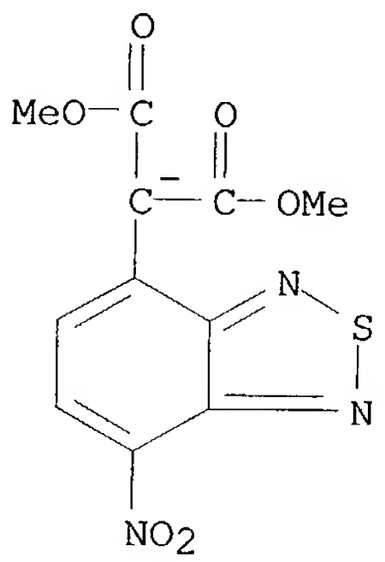
● Na

RN 460723-15-3 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-acetonitrile, α-(2,2-dimethyl-1-oxopropyl)-7-nitro-, ion(1-), sodium (9CI) (CA INDEX NAME)



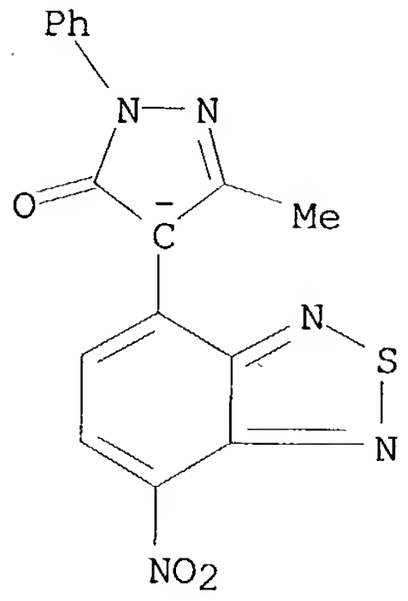
● Na^+

RN 460723-16-4 HCAPLUS
CN Propanedioic acid, (7-nitro-2,1,3-benzothiadiazol-4-yl)-, dimethyl ester, ion(1-), sodium (9CI) (CA INDEX NAME)



● Na^+

RN 460723-17-5 HCAPLUS
CN 3H-Pyrazol-3-one, 2,4-dihydro-5-methyl-4-(7-nitro-2,1,3-benzothiadiazol-4-yl)-2-phenyl-, ion(1-), sodium (9CI) (CA INDEX NAME)



● Na^+

RN 460723-18-6 HCAPLUS
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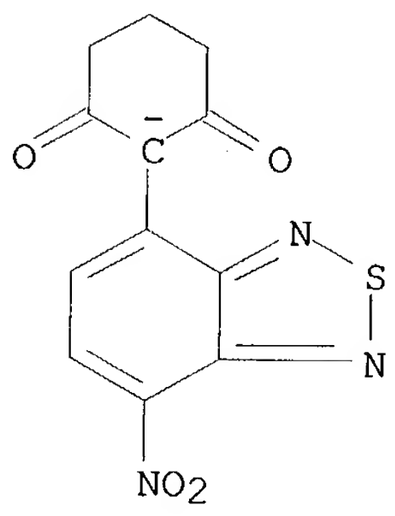
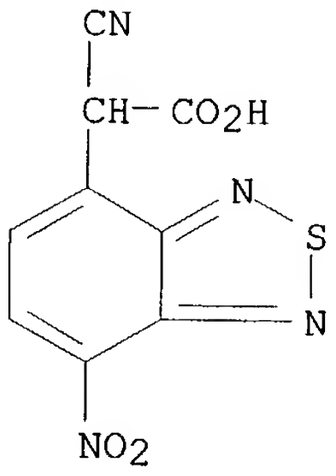


● Na^+

RN 460723-19-7 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-acetic acid, α-cyano-7-nitro-, sodium salt (9CI) (CA INDEX NAME)



● Na

RN 460723-20-0 HCAPLUS
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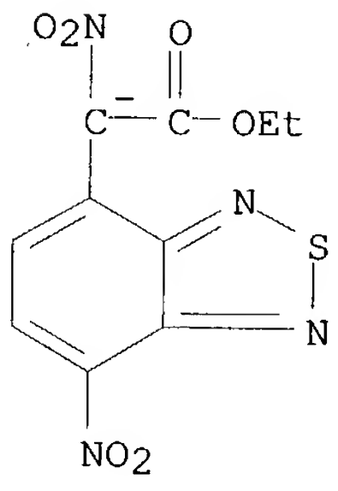
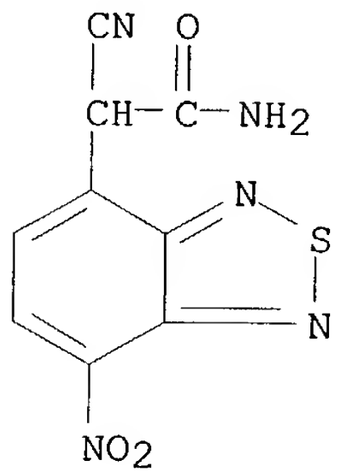


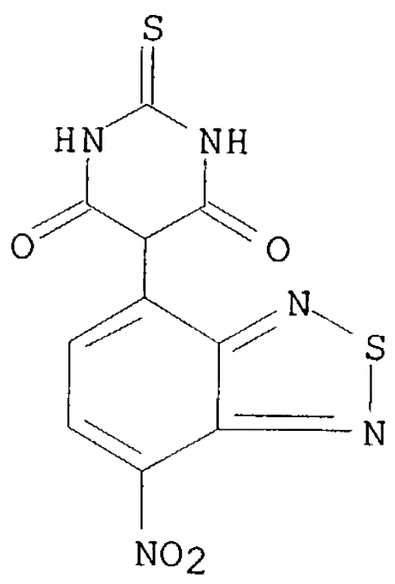
● Na^+

RN 460723-21-1 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-acetamide, α-cyano-7-nitro-, monosodium salt (9CI) (CA INDEX NAME)



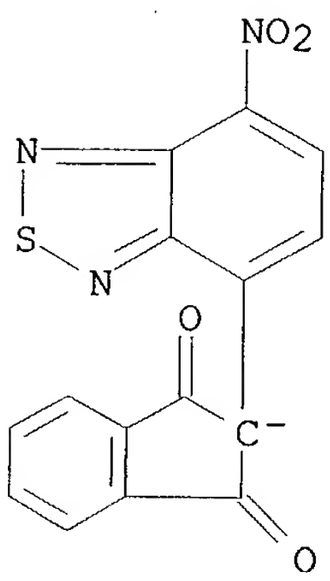
● Na

RN 460723-22-2 HCAPLUS
CN 4,6(1H,5H)-Pyrimidinedione, dihydro-5-(7-nitro-2,1,3-benzothiadiazol-4-yl)-2-thioxo-, monosodium salt (9CI) (CA INDEX NAME)



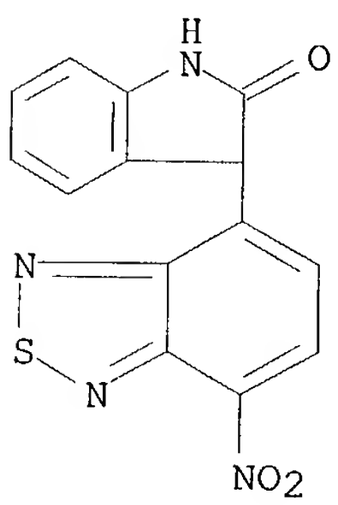
● Na

RN 460723-23-3 HCAPLUS
CN 1H-Indene-1,3(2H)-dione, 2-(7-nitro-2,1,3-benzothiadiazol-4-yl)-, ion(1-), sodium (9CI) (CA INDEX NAME)



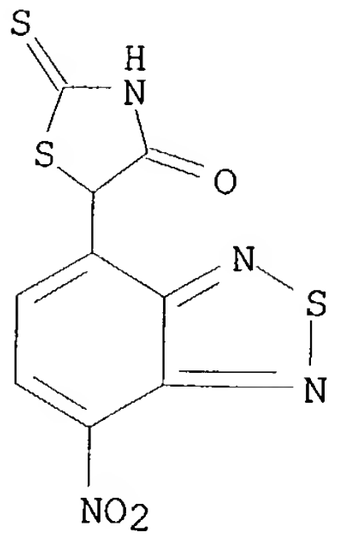
● Na^+

RN 460723-24-4 HCAPLUS
CN 2H-Indol-2-one, 1,3-dihydro-3-(7-nitro-2,1,3-benzothiadiazol-4-yl)-, sodium salt (9CI) (CA INDEX NAME)



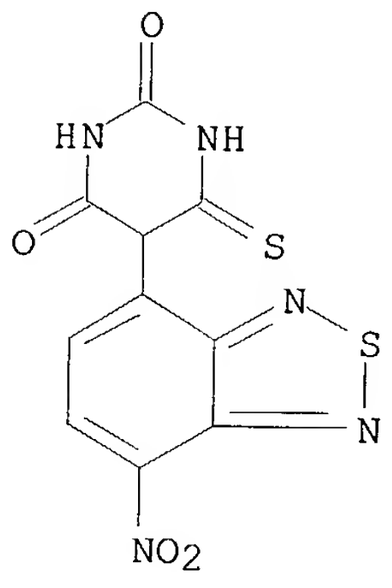
● Na

RN 460723-25-5 HCAPLUS
CN 4-Thiazolidinone, 5-(7-nitro-2,1,3-benzothiadiazol-4-yl)-2-thioxo-, sodium salt (9CI) (CA INDEX NAME)



● Na

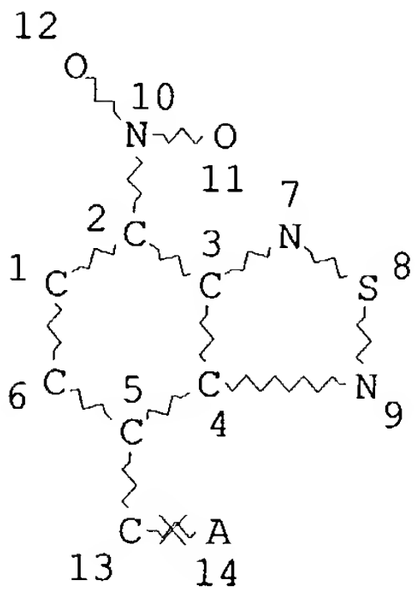
RN 460723-26-6 HCAPLUS
CN 2,4(1H,3H)-Pyrimidinedione, dihydro-5-(7-nitro-2,1,3-benzothiadiazol-4-yl)-6-thioxo-, monosodium salt (9CI) (CA INDEX NAME)



● Na

=> => D QUE
L36 STR

KATHLEEN FULLER EIC 1700 REMSEN 4B28 571/272-2505



NODE ATTRIBUTES:
NSPEC IS RC AT 13
NSPEC IS RC AT 14
DEFAULT MLEVEL IS ATOM
DEFAULT ECLEVEL IS LIMITED

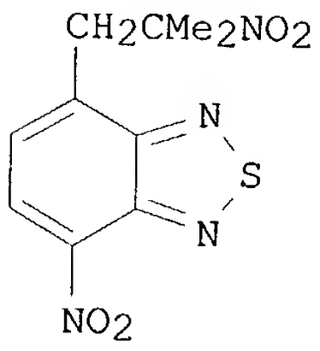
GRAPH ATTRIBUTES:
RING(S) ARE ISOLATED OR EMBEDDED
NUMBER OF NODES IS 14

STEREO ATTRIBUTES: NONE
L38 57 SEA FILE=REGISTRY SSS FUL L36
L40 14 SEA FILE=HCAPLUS ABB=ON L38
L41 1 SEA FILE=HCAPLUS ABB=ON L40 AND (HAIR OR KERAT?)
L42 13 SEA FILE=HCAPLUS ABB=ON L40 NOT L41

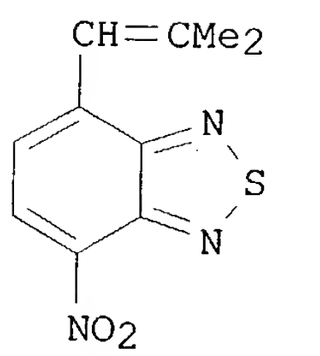
Remaining 13 C A references with no utility

=> D L42 1-13 BIB ABS IND HITSTR

L42 ANSWER 1 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1997:370802 HCAPLUS
DN 127:95244
TI An original way for synthesis of new nitrobenzothiadiazole derivatives
AU Vanelle, Patrice; Liegeois, Celine Tremblais; Meuche, Jacobine; Maldonado, Jose; Crozet, Michel P.
CS Lab. Chim. Org., Fac. Pharm., Univ. Aix-marseille 2, Marseille, 13385, Fr.
SO Heterocycles (1997), 45(5), 955-962
CODEN: HTCYAM; ISSN: 0385-5414
PB Japan Institute of Heterocyclic Chemistry
DT Journal
LA English
OS CASREACT 127:95244
GI



I



II

AB The C-alkylation reaction of 4-chloromethyl-7-nitro-2,1,3-benzothiadiazole with 2-nitropropane anion (which is shown to proceed by an SRN1 mechanism) is an original way for the synthesis of new 2,1,3-benzothiadiazoles I and II.

CC 28-10 (Heterocyclic Compounds (More Than One Hetero Atom))

ST nitrobenzothiadiazole prepn; benzothiadiazole nitro prepn

IT 79-46-9, 2-Nitropropane 570-24-1 3958-63-2
RL: RCT (Reactant); RACT (Reactant or reagent)
(preparation of nitrobenzothiadiazoles)

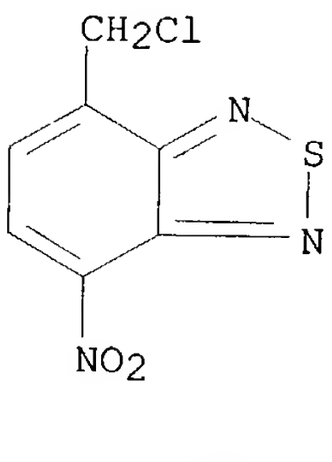
IT 1457-92-7P 2687-25-4P 5170-68-3P, 2,1,3-Benzothiadiazole-4-carboxaldehyde 16405-99-5P 16406-00-1P, 2,1,3-Benzothiadiazole-4-methanol 19706-16-2P 151869-78-2P **191996-19-7P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of nitrobenzothiadiazoles)

IT **191996-20-0P 191996-21-1P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of nitrobenzothiadiazoles)

IT **191996-19-7P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation of nitrobenzothiadiazoles)

RN 191996-19-7 HCAPLUS

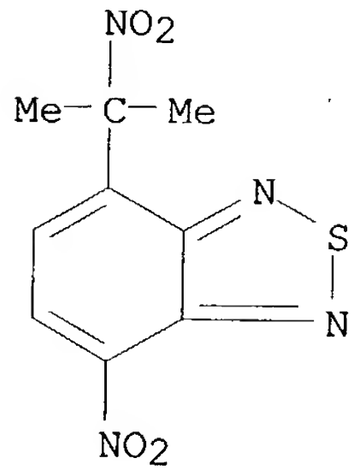
CN 2,1,3-Benzothiadiazole, 4-(chloromethyl)-7-nitro- (9CI) (CA INDEX NAME)



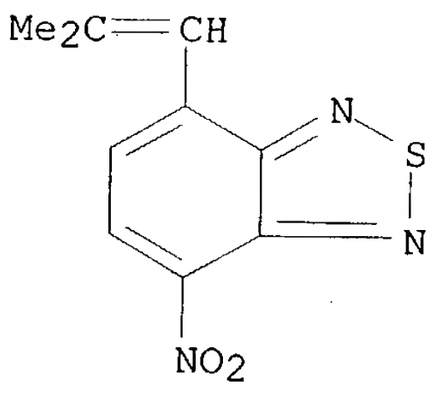
IT **191996-20-0P 191996-21-1P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of nitrobenzothiadiazoles)

RN 191996-20-0 HCAPLUS

CN 2,1,3-Benzothiadiazole, 4-(1-methyl-1-nitroethyl)-7-nitro- (9CI) (CA INDEX NAME)

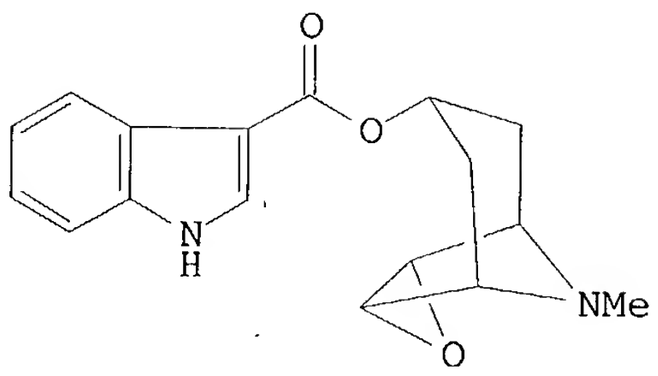


RN 191996-21-1 HCAPLUS
CN 2,1,3-Benzothiadiazole, 4-(2-methyl-1-propenyl)-7-nitro- (9CI) (CA INDEX NAME)

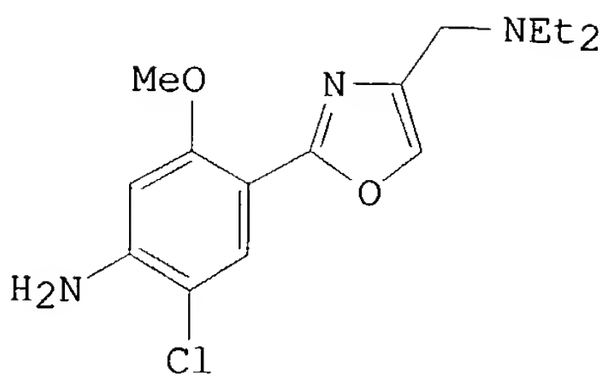


RE.CNT 29 THERE ARE 29 CITED REFERENCES AVAILABLE FOR THIS RECORD
ALL CITATIONS AVAILABLE IN THE RE FORMAT

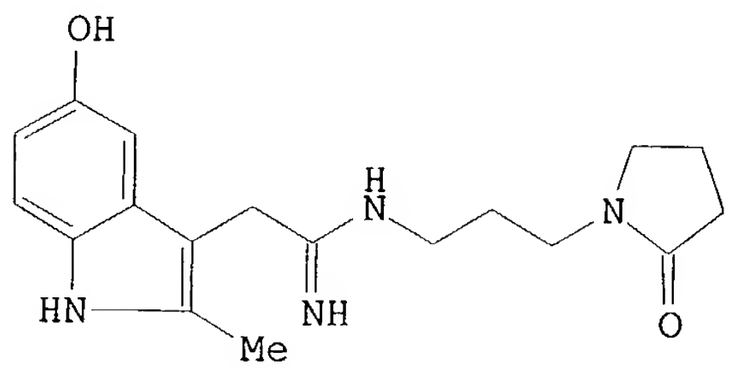
L42 ANSWER 2 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1993:625779 HCAPLUS
DN 119:225779
TI Design and synthesis of novel ligands for the 5-HT3 and the 5-HT4 receptor
AU Blum, E.; Buchheit, K. H.; Buescher, H. H.; Gamse, R.; Kloeppner, E.; Meigel, H.; Papageorgiou, C.; Waelchli, R.; Revesz, L.
CS Preclin. Res., Sandoz Pharma AG, Basel, CH-4002, Switz.
SO Bioorganic & Medicinal Chemistry Letters (1992), 2(5), 461-6
CODEN: BMCLE8; ISSN: 0960-894X
DT Journal
LA English
OS CASREACT 119:225779
GI



I



II



III

AB A novel highly potent 5-HT3 antagonist and Tropisetron analog I is described with an increased efficacy to inhibit cisplatin induced emesis in ferrets. Four novel structural classes of gastroprokinetic benzamide bioisosteres, e.g., II, are presented. 5-HT derivs., e.g., III, are described as ligands of the recently discovered 5-HT4 receptor.

CC 27-11 (Heterocyclic Compounds (One Hetero Atom))
Section cross-reference(s): 1

ST HT receptor indole quinoline deriv; emesis inhibitor tropisetron analog; gastroprokinetic benzamide deriv

IT Neurotransmitter antagonists
(serotoninergic, indole and quinoline derivs.)

IT 570-24-1 150879-82-6 **150879-83-7**
RL: RCT (Reactant); RACT (Reactant or reagent)
(benzothiadiazole derivative from)

IT 501-53-1, Benzyl chloroformate 867-13-0 1006-94-6, 5-Methoxyindole
RL: RCT (Reactant); RACT (Reactant or reagent)
(hydroxyimidazole ketone derivative from)

IT 1076-74-0 150879-84-8 150879-85-9 150879-86-0 150879-87-1
RL: RCT (Reactant); RACT (Reactant or reagent)
(hydroxyindole derivative from)

IT 28957-72-4 117843-63-7 117869-79-1
RL: RCT (Reactant); RACT (Reactant or reagent)
(hydroxyindolyl ketone derivative from)

IT 608-07-1 150879-91-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(hydroxyindolylethylamine derivative from)

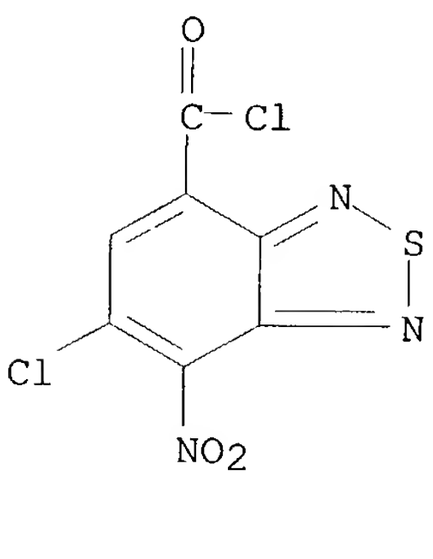
IT 20776-45-8, O-Benzylserotonin
RL: RCT (Reactant); RACT (Reactant or reagent)
(hydroxymetazole derivative from)

IT 6836-19-7 150879-88-2
RL: RCT (Reactant); RACT (Reactant or reagent)
(hydroxynaphthylethylamine derivative from)

IT 41037-26-7 150879-89-3 150879-90-6
RL: RCT (Reactant); RACT (Reactant or reagent)
(hydroxyquinolylethylamine derivative from)

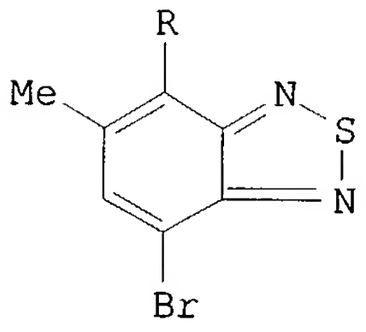
IT 498-45-3, Scopine 771-50-6, 1H-Indole-3-carboxylic acid
RL: RCT (Reactant); RACT (Reactant or reagent)

(indole derivative from)
IT 872-50-4P, N-Methylpyrrolidone, preparation
RL: PREP (Preparation)
(indole quinuclidine derivative from)
IT 4792-58-9 6066-82-6, N-Hydroxysuccinimide 92622-98-5
RL: RCT (Reactant); RACT (Reactant or reagent)
(indole quinuclidine derivative from)
IT 141-97-9, Ethyl acetoacetate 4093-31-6 13324-11-3 15855-37-5
63918-33-2 150879-75-7 150879-76-8 150879-77-9 150879-78-0
150879-79-1
RL: RCT (Reactant); RACT (Reactant or reagent)
(metoclopramide derivative from)
IT 530-62-1 534-07-6, 1,3-Dichloroacetone 7206-70-4 150879-80-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(oxazole derivative from)
IT 364-62-5P 81098-60-4P 90182-92-6P 112727-80-7P 117843-65-9P
122732-06-3P 150879-63-3P 150879-64-4P 150879-65-5P 150879-66-6P
150879-67-7P 150879-68-8P 150879-69-9P 150879-70-2P 150879-71-3P
150879-72-4P 150879-73-5P 150879-74-6P 150880-71-0P 150880-72-1P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation and 5-HT receptor antagonistic activity of)
IT 95-69-2 107-02-8, 2-Propenal, preparation 6238-14-8,
3-Aminoquinuclidine 27527-95-3 150879-81-5
RL: RCT (Reactant); RACT (Reactant or reagent)
(quinoline quinuclidine derivative from)
IT **150879-83-7**
RL: RCT (Reactant); RACT (Reactant or reagent)
(benzothiadiazole derivative from)
RN 150879-83-7 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carbonyl chloride, 6-chloro-7-nitro- (9CI) (CA INDEX NAME)



L42 ANSWER 3 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1979:439391 HCAPLUS
DN 91:39391
TI 1,2,5-Thiadiazole derivatives: Part III. Synthesis and substitution reactions of 4-bromo-6-methylbenzo-2,1,3-thiadiazole and its derivatives
AU Sharma, K. S.; Singh, Vijender; Singh, Ram Phul
CS Chem. Dep., M. D. Univ., Rohtak, India
SO Indian Journal of Chemistry, Section B: Organic Chemistry Including Medicinal Chemistry (1978), 16B(10), 892-4
CODEN: IJSBDB; ISSN: 0376-4699
DT Journal
LA English

OS CASREACT 91:39391
GI



I, R=H

II, R=NO_2

AB The thiadiazole I was prepared from 3-bromo-4,5-diaminotoluene, which in turn was prepared from p-aminotoluene. I was subjected to electrophilic and nucleophilic substitution reactions. Similarly, II was also subjected to nucleophilic substitution reactions giving 7-substitution products replacing the Br.

CC 28-11 (Heterocyclic Compounds (More Than One Hetero Atom))
ST thiadiazole bromo nucleophilic electrophilic substitution
IT Substitution reaction, electrophilic
Substitution reaction, nucleophilic
(of bromobenzothiadiazoles)
IT 110-91-8, reactions
RL: RCT (Reactant); RACT (Reactant or reagent)
(amination of bromobenzothiadiazole by)
IT 7719-09-7
RL: RCT (Reactant); RACT (Reactant or reagent)
(cyclization of, with diaminotoluene, benzothiadiazole derivative from)
IT 614-83-5
RL: RCT (Reactant); RACT (Reactant or reagent)
(nitration of)
IT 70733-34-5P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and cyanation of)
IT 70733-25-4P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and cyclization of, with thionyl chloride, benzothiadiazole derivative from)
IT 70733-24-3P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and deacetylation of)
IT 2450-45-5P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and nucleophilic and electrophilic substitutions of)
IT 70733-29-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and nucleophilic substitution of)
IT 827-24-7P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reduction of)

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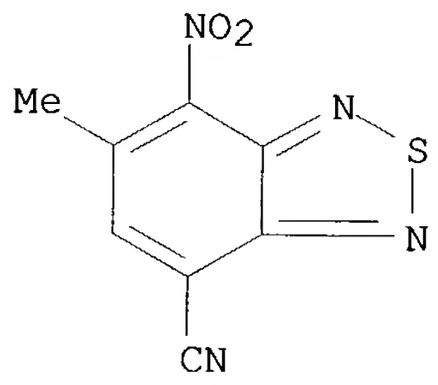
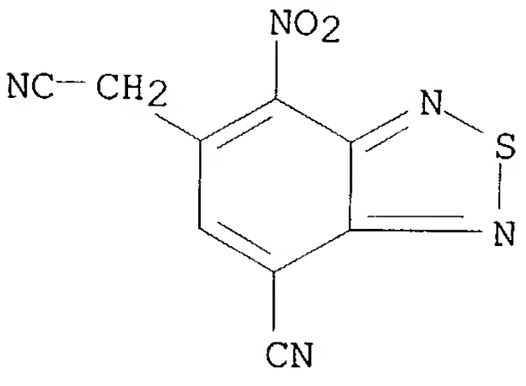
IT 70733-26-5P 70733-27-6P 70733-28-7P 70733-30-1P 70733-31-2P
70733-32-3P **70733-33-4P 70733-35-6P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT **70733-33-4P 70733-35-6P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 70733-33-4 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carbonitrile, 6-methyl-7-nitro- (9CI) (CA INDEX NAME)



RN 70733-35-6 HCAPLUS
CN 2,1,3-Benzothiadiazole-5-acetonitrile, 7-cyano-4-nitro- (9CI) (CA INDEX NAME)



L42 ANSWER 4 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1975:473314 HCAPLUS
DN 83:73314
TI Herbicidal activity of 2,1,3-benzothiadiazolecarbonitriles and related cyanoheterocycles
AU Schieferstein, Robert H.; Pilgram, Kurt
CS Biol. Sci. Res. Cent., Shell Dev. Co., Modesto, CA, USA
SO Journal of Agricultural and Food Chemistry (1975), 23(3), 392-5
CODEN: JAFCAU; ISSN: 0021-8561
DT Journal
LA English
GI For diagram(s), see printed CA Issue.
AB New carbonitriles of 2,1,3-benzothiadiazole and benzofurazan have been prepared and evaluated for herbicidal activity. 4,7- (I) [20138-79-8], 4,5-dicyano-2,1,3-benzothiadiazole [54512-77-5], 4,7- [20138-81-2], and 4,5-dicyanobenzofurazan [54286-60-1] were active pre- and postemergence at low rates. Substitution of 1 or both cyano groups by hydrogen, alkyl, chlorine, carboxy, alkoxycarbonyl, carboxyamido, acylamido, and ureido reduced activity significantly. High activity was maintained in the

monomethyl analog of I, whereas addition of 2 methyl groups or 1 amino or nitro group essentially eliminated activity. Annual ryegrass, wild oat, and corn have tolerance for I in relation to rates required for control of a wide range of weeds; other analogs do not appear as selective for corn as I.

CC 5-3 (Agrochemicals)

ST benzothiadiazole carbonitrile herbicide

IT Herbicides
(benzothiadiazolecarbonitriles and cyanoheterocycles)

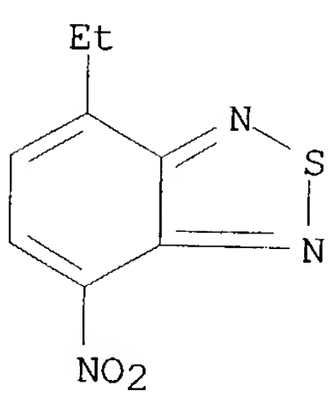
IT Molecular structure-biological activity relationship
(herbicidal, of benzothiadiazolecarbonitriles and cyanoheterocycles)

IT 1982-55-4P 2207-34-3P 2255-96-1P 2325-05-5P 5023-20-1P
5170-41-2P 16100-06-4P 16408-05-2P 20138-79-8P 20138-80-1P
20138-81-2P 20138-82-3P 54286-59-8P 54286-60-1P 54286-62-3P
54512-76-4P 54512-77-5P 54512-78-6P 54512-79-7P 54512-80-0P
54512-81-1P 54512-82-2P 54535-88-5P 54535-90-9P 54535-91-0P
54535-92-1P 54535-93-2P 54535-94-3P **54535-95-4P**
54535-96-5P 54535-97-6P 54535-98-7P **54535-99-8P**
54554-45-9P 54558-20-2P 54558-21-3P 54558-22-4P **54558-24-6P**
54558-25-7P 55921-99-8P 55922-00-4P 55922-01-5P 55922-02-6P
55922-03-7P **55954-31-9P**
RL: AGR (Agricultural use); BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); USES (Uses)
(preparation and herbicidal activity of)

IT **54535-95-4P 54535-99-8P 54558-24-6P**
55954-31-9P
RL: AGR (Agricultural use); BAC (Biological activity or effector, except adverse); BSU (Biological study, unclassified); SPN (Synthetic preparation); BIOL (Biological study); PREP (Preparation); USES (Uses)
(preparation and herbicidal activity of)

RN 54535-95-4 HCAPLUS

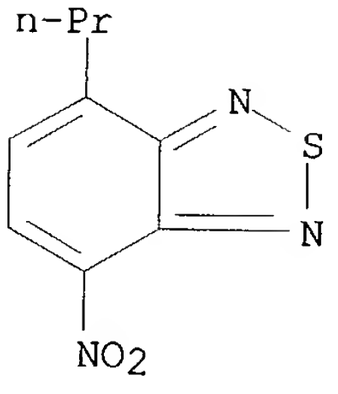
CN 2,1,3-Benzothiadiazole, 4-ethyl-7-nitro- (9CI) (CA INDEX NAME)



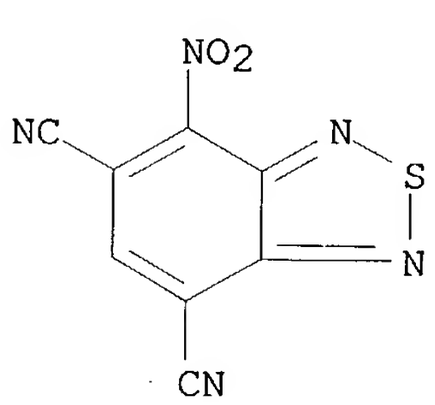
RN 54535-99-8 HCAPLUS

CN 2,1,3-Benzothiadiazole, 4-nitro-7-propyl- (9CI) (CA INDEX NAME)

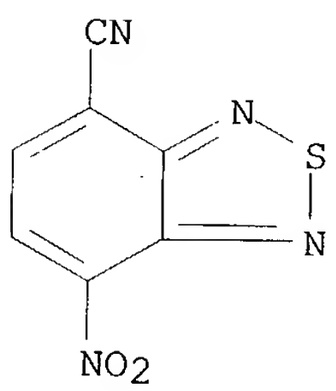
KATHLEEN FULLER EIC 1700 REMSEN 4B28 571/272-2505



RN 54558-24-6 HCAPLUS
CN 2,1,3-Benzothiadiazole-4,6-dicarbonitrile, 7-nitro- (9CI) (CA INDEX NAME)

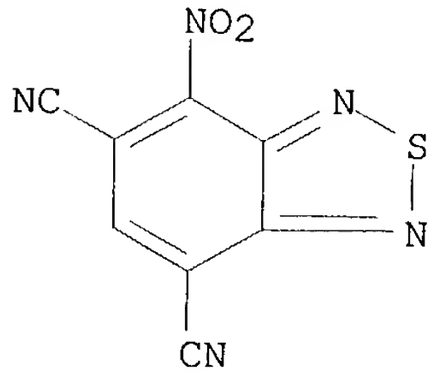


RN 55954-31-9 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carbonitrile, 7-nitro- (9CI) (CA INDEX NAME)



L42 ANSWER 5 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1975:43278 HCAPLUS
DN 82:43278
TI Synthesis of 2,1,3-benzothiadiazolecarbonitriles
AU Pilgram, K.; Skiles, R. D.
CS Biol. Sci. Res. Cent., Shell Dev. Co., Modesto, CA, USA
SO Journal of Heterocyclic Chemistry (1974), 11(5), 777-80
CODEN: JHTCAD; ISSN: 0022-152X
DT Journal
LA English
OS CASREACT 82:43278
AB 2,1,3-Benzothiadiazolemono- and dicarbonitriles (I) were prepared by reaction of bromo-2,1,3-benzothiadiazoles with CuCN in refluxing DMF to give I, complexed with CuBr. H2O2 in HCl at 30-40° decomposed these complexes. Yields in the Sandmeyer method for preparing nitriles I were improved by diazotizing amino-2,1,3-benzothiadiazoles with

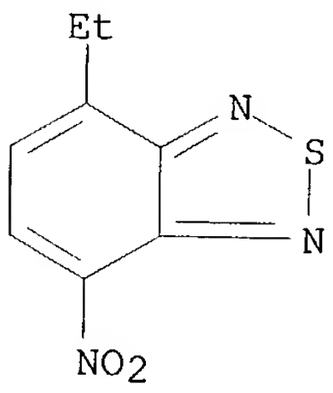
nitrosylsulfuric acid prior to reaction with CuCN-NaCN.
CC 28-11 (Heterocyclic Compounds (More Than One Hetero Atom))
ST benzothiadiazolecarbonitrile; nitrile benzothiadiazolyl; Sandmeyer aminobenzothiadiazole
IT Sandmeyer reaction
(of aminobenzothiadiazoles)
IT 874-37-3
RL: RCT (Reactant); RACT (Reactant or reagent)
(bromination of)
IT 18392-81-9P 54558-23-5P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reaction of, with cyanide)
IT 49764-63-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reaction of, with sulfinylaniline)
IT 20138-79-8P 54512-76-4P 54512-77-5P 54512-78-6P 54512-79-7P
54512-80-0P 54512-81-1P 54512-82-2P 54554-45-9P 54558-20-2P
54558-21-3P 54558-22-4P **54558-24-6P** 54558-25-7P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 767-64-6 874-37-3 2255-79-0 2255-80-3 2255-81-4 2274-65-9
15155-41-6 16407-86-6 18392-74-0 28681-43-8 28681-49-4
54558-26-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with cyanide)
IT 54558-19-9
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with dibromophenylenediamine)
IT 54558-18-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(reduction of)
IT **54558-24-6P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 54558-24-6 HCAPLUS
CN 2,1,3-Benzothiadiazole-4,6-dicarbonitrile, 7-nitro- (9CI) (CA INDEX NAME)



L42 ANSWER 6 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1975:43273 HCAPLUS
DN 82:43273
TI 4,7-Disubstituted 2,1,3-benzothiadiazoles
AU Pilgram, K.
CS Biol. Sci. Res. Cent., Shell Dev. Co., Modesto, CA, USA
SO Journal of Heterocyclic Chemistry (1974), 11(5), 835-7

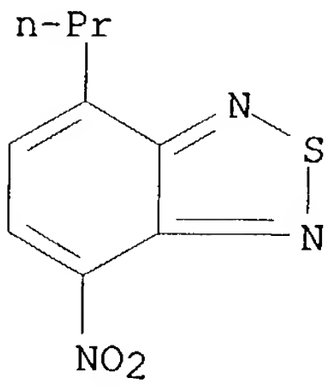
KATHLEEN FULLER EIC 1700 REMSEN 4B28 571/272-2505

CODEN: JHTCAD; ISSN: 0022-152X
DT Journal
LA English
GI For diagram(s), see printed CA Issue.
AB Benzothiadiazoles I (R = R1 = Me, CO2H, CO2Me, CO2Et, CONH2, CONHMe, CONHNH2; R = Et, R1 = NO2, NH2, NHCONHMe, NHAc, NHCOCH2Cl; R = NMe2, R1 = NO2, NH2; R = Me, R1 = NHCONHMe, NHCONMe2) were prepared Thus 2,5-Me2C6-H3NH2 was nitrated and the 2,5,6-Me2(O2N)C6H2NH2 reduced to the diamine and treated with N-sulfinylaniline to give I (R = R1 = Me).
CC 28-11 (Heterocyclic Compounds (More Than One Hetero Atom))
ST benzothiadiazole; xylidine nitration; aminoxylene reaction sulfinylaniline
IT 26460-78-6 54535-91-0
RL: RCT (Reactant); RACT (Reactant or reagent)
(amination of)
IT 20138-79-8
RL: RCT (Reactant); RACT (Reactant or reagent)
(hydrolysis of)
IT 95-78-3 17754-04-0
RL: RCT (Reactant); RACT (Reactant or reagent)
(nitration of)
IT 54535-89-6P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and esterification of)
IT 3171-46-8P
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reaction of, with sulfinylaniline)
IT 15540-85-9P 54535-93-2P **54535-95-4P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reduction of)
IT 2325-05-5P 5170-41-2P 54535-88-5P 54535-90-9P 54535-92-1P
54535-94-3P 54535-96-5P 54535-97-6P 54535-98-7P **54535-99-8P**
54536-00-4P 54536-01-5P 54536-02-6P 54536-03-7P 54536-04-8P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT 1122-83-4
RL: RCT (Reactant); RACT (Reactant or reagent)
(reaction of, with dimethylphenylenediamine)
IT **54535-95-4P**
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)
(preparation and reduction of)
RN 54535-95-4 HCAPLUS
CN 2,1,3-Benzothiadiazole, 4-ethyl-7-nitro- (9CI) (CA INDEX NAME)



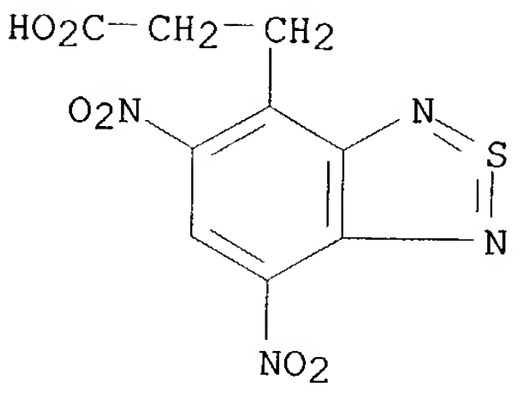
KATHLEEN FULLER EIC 1700 REMSEN 4B28 571/272-2505

IT **54535-99-8P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 54535-99-8 HCAPLUS
CN 2,1,3-Benzothiadiazole, 4-nitro-7-propyl- (9CI) (CA INDEX NAME)



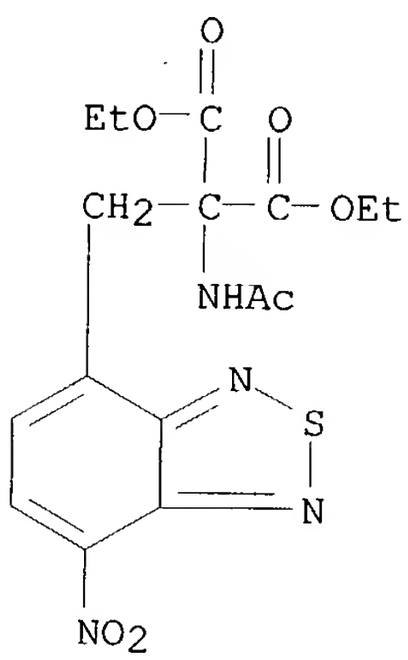
L42 ANSWER 7 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1970:31707 HCAPLUS
DN 72:31707
TI 2,1,3-Thia- and selenadiazoles. LIX. Carboxy-, carboxymethyl-, and carboxyethylbenzo-2,1,3-thiadiazoles
AU Pesin, V. G.; D'yachenko, S. A.; Golubeva, E. V.
CS Leningrad. Khim.-Farm. Inst., Leningrad, USSR
SO Khimiya Geterotsiklicheskikh Soedinenii (1969), (4), 619-22
CODEN: KGSSAQ; ISSN: 0132-6244
DT Journal
LA Russian
GI For diagram(s), see printed CA Issue.
AB To a solution of 0.23 g Na in 15 ml anhydrous EtOH was added 1.6 g di-Et malonate, the mixture stirred 1 hr, and 2.29 g I (R = Br) in 25 ml dry C6H6 added, and the whole kept 10 hr to yield 81% I (R = CH2CO2H), m. 103-4° (H2O). To a solution of 0.92 g Na in 40 ml EtOH was added 6.4 g di-Et malonate, the mixture stirred 1 hr, and 9.2 g II (R = Br) in 80 ml dry EtOH added to yield 29% III (R = R1 = CO2Et) (IV), m. 105-6° (EtOH), and, from the mother liquor (after 8-10 hr reflux with 120 ml 20% HCl) 4.8 g II (R = CH2CO2H), m. 117-18° (H2O). IV (2 g) in 40 ml 10% KOH was refluxed 3 hr to give 90% III (R = H, R1 = CO2H), m. 141-2° (EtOH). To 10 ml HNO3 (d. 1.5) was added dropwise with stirring 1 g II (R = CO2H) and the mixture kept 30 min at 20° to give 73% V (R = CO2H), m . 180-2° (EtOH). To a solution of 1.5 g KCN in 75 ml EtOH and 5 ml H2O was added portionwise 2 g I (R = Br) and the whole refluxed 3 hr to yield 0.6 g VII (R = CN) (VIII), m. 192-3° (AcOH), and 1 g I (R = CN), m. 92 -3° (EtOH). VIII (1 g) in 25 ml 50% H2SO4 and 25 ml AcOH was refluxed 3 hr to yield 95% VII (R = CO2H), m. 179-80° (EtOH). To 12 ml HNO3 (d 1.5) was added portionwise at 0° during 30 min 1 g II (R = CH2CO2H), and the mixture stirred 30 min and poured on ice to yield 85% V (R = CH2CO2H), m. 153-4° (aqueous EtOH). To 15 ml HNO3 (d. 1.5) was added portionwise at 0° with stirring 1.5 g. I (R = CH2CO2H) to yield 75% VI (R = CH2CO2H), m. 137-8° (EtOH). The pK values of the acids obtained were measured and compared with those of the corresponding aromatic carboxylic acids.
CC 28 (Heterocyclic Compounds (More Than One Hetero Atom))
ST benzothiadiazoles; thiadiazoles benzo
IT Propionic acid, 2,3-di-2,1,3-benzothiadiazol-4-yl-
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)

IT 16406-01-2P, 2,1,3-Benzothiadiazole-4-acetonitrile 24786-02-5P
24786-03-6P 24786-04-7P 24786-05-8P 24786-06-9P 24786-07-0P
24786-10-5P **24786-11-6P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT **24786-11-6P**
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
RN 24786-11-6 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-propionic acid, 5,7-dinitro- (8CI) (CA INDEX NAME)



L42 ANSWER 8 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1968:496583 HCAPLUS
DN 69:96583
TI 2,1,3-thia- and selenadiazoles. LII. Derivatives of β-phenylalanine
AU Pesin, V. G.; D'yachenko, S. A.
CS Leningrad. Khim.-Farm. Inst., Leningrad, USSR
SO Khimiya Geterotsiklicheskikh Soedinenii (1968), (2), 254-5
CODEN: KGSSAQ; ISSN: 0132-6244
DT Journal
LA Russian
GI For diagram(s), see printed CA Issue.
AB From appropriate derivs. of 2,1,3-thiadiazoles and the Na salt of acetamidomalonic ester were obtained: 92% 4-(β,β-dicarbethoxy-β-acetamidoethyl)-2,1,3-benzothiadiazole (I), m. 118-19° (EtOH-H2O), 69% 5-(β,β-dicarbethoxy-β-acetamidoethyl)-2,1,3-benzothiadiazole (II), m. 132-3° (EtOH); 91% 4-(β,β-dicarbethoxy)-β-acetamidoethyl)-7-nitro-2,1,3-benzothiadiazole (III) m. 205° (EtOH). In acidic solution I-III were decarboxylated to give 4-(β-carboxy-β-aminoethyl)-2,1,3-benzothiadiazole (IV), m. 283-4° [hydrochloride m. 216° (EtOH-Et2O)]; 5-(β-carboxy-β-aminoethyl)-2,1,3-benzothiadiazole, m. 278° (H2O) [hydrochloride m. 250° (EtOH-Et2O)]; 4-(β-carboxy-β-aminoethyl)-7-nitro-2,1,3-benzothiadiazole-HCl, m. 217° (EtOH-Et2O). IV heated in 50% EtOH at 80° with salicylaldehyde gave 60% 4-[β-carboxy-β-(aminosalicylideneaminoethyl]-2,1,3-benzothiadiazole, m. 312-14°.
CC 28 (Heterocyclic Compounds (More Than One Hetero Atom))
ST benzothiadiazoles; thiadiazoles benzo; phenylalanine derivs
IT **7196-36-3P** 20032-76-2P 20032-77-3P 20032-78-4P 20032-79-5P
20032-80-8P 20032-81-9P 20361-50-6P
RL: SPN (Synthetic preparation); PREP (Preparation)
(preparation of)
IT **7196-36-3P**
RL: SPN (Synthetic preparation); PREP (Preparation)

(preparation of)
RN 7196-36-3 HCAPLUS
CN Malonic acid, acetamido[(7-nitro-2,1,3-benzothiadiazol-4-yl)methyl]-, diethyl ester (7CI, 8CI) (CA INDEX NAME)



NO_2

L42 ANSWER 9 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1966:465498 HCAPLUS
DN 65:65498
OREF 65:12196b-g
TI Chemistry of 1,2,3-thia- and -selenadiazoles. XL. Bis(βchloroethyl) amino derivatives
AU Pesin, V.G.; D'yach-enko, S. A.
CS Chem.-Pharm. Inst., Leningrad
SO Khimiya Geterotsiklicheskikh Soedinenii (1966), (3), 382-6
CODEN: KGSSAQ; ISSN: 0132-6244
DT Journal
LA Russian
AB cf. CA 64, 19595h. A series of 4-(β,β-dicarbethoxy-β-acetylaminoethyl)-7-substituted-2,1,3-benzothiadiazoles (I)and a series of 5-(β-carbmethoxy-β-phthalimidoethyl)-4-substituted-2,1,3-benzothiadiazoles (II) were prepared Thus, 5.5 g. AcNHCH(CO2Et)2 (III) was added to a solution of 0.58 g. Na in 30 cc.. EtOH at 45-50°, the mixture was stirred 1 hr. at room temperature, a solution of 6.9 g. 4-bromomethyl-7-nitro-2,1,3-benzothiadiazole (IV) in 60 cc. C6H6 added, and the mixture stirred at room temperature 3 hrs. to give 9.5 g. I (R = NO2) (Ia), m. 205° (EtOH). Similarly, 5-(ββ-dicarbethoxy-β-acetylaminoethyl)-4-nitro-2,1,3-benzotbiadiazole (V), m. 163-4° (EtOH), was prepared from 5-bromomethyl-4nitro-2,1,3-benzothiadiazole (VI), in 75% yield. A mixture of 4.5 g. Ia, 120 cc.. EtOH, 40 cc. H2O tremain, 3.6 cc. AcOH, and 7 g. Fe shavings heated with vigorous stirring on a water bath 2 hrs. gave 3.5 g. I (R = NH2) (Ib), m. 164.5-5.0° (EtOH). A mixture of 0.5 g. Ib, 20 cc. 25% AcOH, and 5 cc. ethylene oxide was kept at room temperature over 2 days, ethylene oxide was distilled, the residue was neutralized with a saturated NaHCO3 solution, and the precipitate dissolved in CHCl3 was placed on a column of Al2O3. From the upper layer was isolated I (R = NHCH2CH2OH) (Ic), from the middle layer unchanged Ib, and from the lower layer I [R = N(CH2CH2OH)2] (Id), m, 131° (AcOEt-petr. ether). Ic (3 g.) was added in small portions to 15 cc. POCl3, the mixture was heated 2 hrs. to

50°, poured onto ice, filtered, basified with NaHCO3, and extracted with CHCl3 to give 0.4 g. I (R = ethylenimino) (Ie), m. 73-4° (C6H6-petr. ether). The structure of Ie was only suggested on the basis Of its ir spectrum. Freshly distilled ethylene oxide (6 cc..) added to a mixture of 3.5 g. Ib and 25 cc. 25% AcOH at 10° and the mixture kept 100 hrs. gave 2.7 g. Id, m. 134° (H2O). Similarly was prepared 58% II [R = N(CH2CH2OH)2] (IIa), m. 141-2° (aqueous EtOH), from II (R = NH2) (IIb). A mixture of 2 g. Id and 10 g. POCl3, heated 2 hrs. at 50-60°, poured onto ice, and extracted with CHCl3, gave 0.8 g. I (R = N(CH2CH2Cl)2) (If), m. 61-2° (aqueous EtOH). Similarly was prepared 51% II [R = N(CH2CH2Cl)2] (IIc),m.98-100° (EtOH),from IIa. Boiling 1.2g. If with 60 cc.. 20% HCl 8 hrs. gave 0.7 g. 4-(β-amino-β-carboxyethyl)-7bis (β-chloroethyl)amino-2,1,3-benzothiadiazole hydrochloride (VII), m. 161-8° (decomposition) (EtOH). Boiling a mixture of 9 g. V and 200 cc. 20% HCl 18 hrs. gave 6 g. 5-(β-amino-β-carboxyethyl)-4-nitro-2,1,3-benzothiadiazole hydrochloride (VIII), m. 270° (Et2O-EtOH). A mixture of 1.6 g. VIII, 0.8 g. phthalic anhydride, and 16 cc. C5H5N was heated 2 hrs. at 80°, C5H5N distilled, the residue heated with 2.5 cc.. Ac2O 1 hr. at 80° and poured into H2O, and the precipitate was dissolved in 20 cc. MeOH and saturated with HCl at 60° to give 2.2 g. II (R = NO2) (IId), m. 199-200° (MeOH). A mixture of 10 cc. H2O, 2 cc. AcOH, and 1.5 g. reduced Fe added to a hot solution of 3.5 g. IId in 50 cc. dioxane and the mixture heated on a boiling water bath 1.5 hrs. gave 2.3 g. IIb, m. 161-1.5° (EtOH). Treating 3.5 g. IIc with 80 cc. 20% HCl gave 0.8 g. 5-(β-amino-β-carboxyethyl)-4-bis(βchloroethyl)amino-2,1,3-benzothiadiazole hydrochloride, m. 188° (decomposition)(EtOH).

CC 38 (Heterocyclic Compounds (More Than One Hetero Atom))

IT 2,1,3-Benzothiadiazolo-5-propionic acid, 4-amino-α-phthalimido-, methyl ester

IT 273-13-2, 2,1,3-Benzothiadiazole 273-15-4, 2,1,3-Benzoselenadiazole (derivs.)

IT 7185-97-9, Malonic acid, acetamido[(7-amino-2,1,3-benzothiadiazol-4-yl)methyl]-, diethyl ester **7196-36-3**, Malonic acid, acetamido[(7-nitro-2,1,3-benzothiadiazol-4-yl)methyl]-, diethyl ester 7196-37-4, Malonic acid, acetamido[(4-nitro-2,1,3-benzothiadiazol-5-yl)methyl]-, diethyl ester 7229-02-9, Malonic acid, acetamido[[7-(1-aziridinyl)-2,1,3-benzothiadiazol-4-yl]methyl]-, diethyl ester 7229-03-0, Malonic acid, acetamido[[7-[bis(2-chloroethyl)amino]-2,1,3-benzothiadiazol-4-yl]methyl]-, diethyl ester 7229-04-1, 2,1,3-Benzothiadiazolo-5-propionic acid, 4-[bis(2-chloroethyl)amino]-α-phthalimido-, methyl ester 7229-05-2, 2,1,3-Benzothiadiazole-4-alanine, 7-[bis(2-chloroethyl)amino]-, hydrochloride 7229-07-4, 2,1,3-Benzothiadiazole-5-propionic acid, 4-nitro-α-phthalimido-, methyl ester 7229-09-6, 2,1,3-Benzothiadiazole-5-alanine, 4-[bis(2-chloroethyl)amino]-, hydrochloride 7229-49-4, Malonic acid, acetamido[[7-[bis(2-hydroxyethyl)amino]-2,1,3-benzothiadiazol-4-yl]methyl]-, diethyl ester 7263-29-8, 2,1,3-Benzothiadiazole-5-propionic acid, 4-[bis(2-hydroxyethyl)amino]-α-phthalimido-, methyl ester 7288-36-0, Malonic acid, acetamido[[7-[(2-hydroxyethyl)amino]-2,1,3-benzothiadiazol-4-yl]methyl]-, diethyl ester 92660-11-2, 2,1,3-Benzothiadiazole-5-alanine, 4-amino-, hydrochloride
(preparation of)

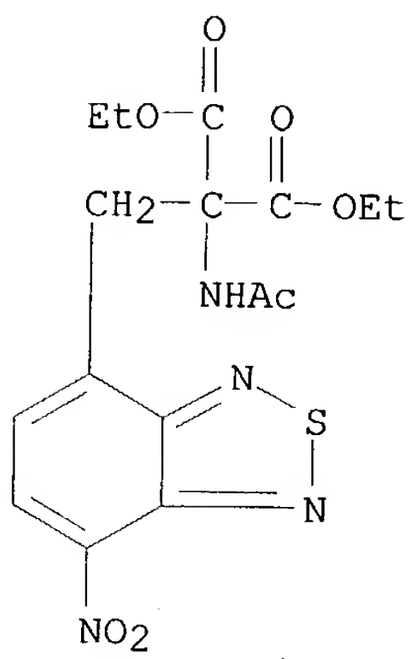
IT **7196-36-3**, Malonic acid, acetamido[(7-nitro-2,1,3-benzothiadiazol-4-yl)methyl]-, diethyl ester
(preparation of)

RN 7196-36-3 HCAPLUS

CN Malonic acid, acetamido[(7-nitro-2,1,3-benzothiadiazol-4-yl)methyl]-,

KATHLEEN FULLER EIC 1700 REMSEN 4B28 571/272-2505

diethyl ester (7CI, 8CI) (CA INDEX NAME)



L42 ANSWER 10 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1965:480630 HCAPLUS
DN 63:80630
OREF 63:14851b-d
TI Studies on 2,1,3-thia- and selenadiazole. XXXVII. The interaction of aromatic o-diamines with thionyl chlorides or thionylaniline
AU Pesin, V. G.; Muravnik, R. S.
CS Chem. Pharm. Inst., Leningrad
SO Latvijas PSR Zinatnu Akademijas Vestis, Kimijas Serija (1965), (2), 233-6
CODEN: LZAKAM; ISSN: 0002-3248
DT Journal
LA Russian
AB Aromatic o-diamines react with SOCl2 or PhNSO forming o-thionylaminoanilines and o-dithionylarylenediamines as intermediate products. These are then transformed into derivs, of benzo-2,1,3-thiadiazole. Equal amts. of aromatic o-diamines and SOCl2 or PhNSO in the presence of anhydrous AlCl3, gave benzo-2,1,3-thiadiazole and some of its derivs, in good yields. Anhydrous AlCl3 (13.5 g.) was dissolved with stirring in 120 ml. pyridine. The solution was cooled to 30° and 10.8 g. o-phenylenediamine (or its salts) added. Then, 12 g. SOCl2 or 13 g. PhNSO was slowly dropped at such a rate that the temperature remained between 35-45°. The mixture was acidified with HCl and steam-separated, the benzo-2,1,3-thiadiazole distd, or filtered off and washed with cold H2O; yield 10.9-11.15 g., m. 43-4°. 4-Methyl- and 5-methylbenzo-2,1,3-thiadiazole, and 1',2'-naphtho-2,1,3-thiadiazole were obtained similarly. 4-Methylbenzo-2,1,3-thiazole b. 229.5-230.5°, d20 1.2448, n20D 1.6265. 5-Methylbenzo-2,1,3-thiazole m. 34°, b. 233-4°. 1',2'-Naphtho-2,1,3-thiazole forms crystals, m. 81° (EtOH).
CC 38 (Heterocyclic Compounds (More Than One Hetero Atom))
IT Amines
(reactions of di-, with N-sulfinylaniline and thionyl chloride)
IT 233-68-1, Naphtho[1,2-c][1,2,5]thiadiazole 273-13-2, 2,1,3-Benzothiadiazole 1457-92-7, 2,1,3-Benzothiadiazole, 4-methyl- 1457-93-8, 2,1,3-Benzothiadiazole, 5-methyl- 3436-82-6, Benzofuran, 5,5'-(1,2-diethylethylene)bis[2-methyl- **3529-18-8**, 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro- 3529-33-7, 2,1,3-Benzothiadiazole-4-sulfonanilide, 7-methyl- 3529-34-8, 2,1,3-Benzothiadiazole-4-sulfonamide, 7-methyl-N-(α-methylphenethyl)-

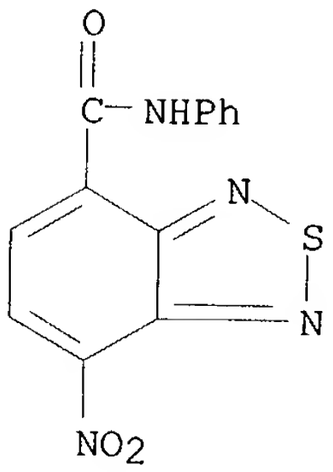
3529-35-9, 2,1,3-Benzothiadiazole-4-sulfonamide, 5-methyl- 3529-36-0, 2,1,3-Benzothiadiazole-4-sulfonamide, N,5-dimethyl- 3529-37-1, 2,1,3-Benzothiadiazole-4-sulfonanilide, 5-methyl- 3529-38-2, 2,1,3-Benzothiadiazole-4-sulfonamide, 5-methyl-N-(α-methylphenethyl)- 3663-15-8, 2,1,3-Benzothiadiazole-4-sulfonamide, 7-methyl- 3746-14-3, 2,1,3-Benzothiadiazole-4-sulfonamide, N,7-dimethyl-
(preparation of)

IT 1122-83-4, Aniline, N-sulfinyl- 7719-09-7, Thionyl chloride
(reaction with aromatic o-diamines)

IT **3529-18-8**, 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro-
(preparation of)

RN 3529-18-8 HCAPLUS

CN 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro- (7CI, 8CI) (CA INDEX NAME)



L42 ANSWER 11 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN

AN 1965:480629 HCAPLUS

DN 63:80629

OREF 63:14850f-h,14851a-b

TI Studies on 2,1,3-thia- and selenadiazole. XXXVI. Sulfonation and oxidation

AU Pesin, V. G.; Muravnik, R. S.

CS Chem. Pharm. Inst., Leningrad

SO Latvijas PSR Zinatnu Akademijas Vestis, Kimijas Serija (1965), (2), 223-32
CODEN: LZAKAM; ISSN: 0002-3248

DT Journal

LA Russian

AB cf. CA 63, 4279c. Sulfonation and oxidn, of methyl derivs, of benz-2,1,3-thiadiazole (I) have been studied. On heating 4-methylbenzo-2,1,3-thiadiazole (II) with 20% oleum at 120-30° for 2 hrs. forms mainly 4-methylbenzo-2,1,3-thiadiazole-7-sulfonic acid (III) which is extremely hygroscopic. 5-Methylbenzo-2,1,3-thiadiazole (IV) in analogous conditions forms 5-methylbenzo-2,1,3-thiadiazole-4-sulfonic acid (V) in 77% yield, m. 202-3°. Structures of III and V were established by converting them into the corresponding Br derivs. of known structures. II or the Na salt of III with chlorosulfonic acid at 150-60° for 1.5 hrs. gave 4-methylbenzo-2,1,3-thiadiazole -7-sulfochloride (VI), m. 134-5°, which with NH3 or amines gave the corresponding amides (VII), and with alcs. gave ester (VIII) (Me and Pr). Analogously, from IV or V 5-methylbenzo-2,1,3-thiadiazole 4-sulfochloride (IX) (m. 152-3°) was obtained which was similarly converted into amides (X) and esters (XI) (Me and Et). On reduction of the sulfochloride VI with Na sulfite, the corresponding sulfinic acid (XII) was obtained in 56% yield, m. 157.5-8.5°. Oxidation of II with chromic acid formed a number of substances from which benzo-2,1,3-thiadiazole-4-carboxylic acid

(XIII) (structure not estimated) could be obtained in 3% yield. 4-Methyl-7-nitrobenzo-2,1,3-thiadiazole (XIV) and 5-methyl-4-nitrobenzo-2,1,3-thiadiazole (XV) on oxidn, with chromic anhydride in the presence of H2SO4 and AcOH, gave correspondingly 7-nitrobenzo-2,1,3-thiadiazole-4-carboxylic acid (XVI), m. 237-8°; and 4-nitrobenzo-2,1,3-thiadiazole-5-carboxylic acid (XVII), m. 245-7°, in 77 and 15% yields, resp. In these conditions 7-chloro-4-methylbenzo-2,1,3-thiadiazole (XVIII) and 7-bromo-4-methylbenzo-2,1,3-thiadiazole (XIX) gave 7-chlorobenzo-2,1,3-thiadiazole-4-carboxylic acid (XX) (m. 254-5°) and 7-bromobenzo-2,1,3-thiadiazole-4-carboxylic acid (XXI) (m. 216-17°), resp., in 39 and 52% yields. XVI with SOCl2 gave the corresponding acid chloride (XXII), m. 94.5-96°, which with alcs. or dimers gave the corresponding esters (XXIII) and amides (XXIV). Reduction of XXII (Et or diethylaminoethyl ester) gave the corresponding amines (XXV) and (XXVI), analogous to anesthesine and monocaine with a thiadiazole ring. Acid XVII with SOCl2 similarly gave the corresponding acid chloride (XXVII) which gave the anilide (XXVIII) on treatment with an ethereal solution of aniline. XXVIII was obtained in 60% yield, m. 217-18°. XXV was obtained in 35% yield, m. 149-50°. XXVI was obtained as hydrochloride in 25% yield, m. 214-15°. Yields and m.ps. of a number of amides (VII, X, XXIV) and esters (VIII, XI, XXIII) of acids III, V, and XVI are tabulated.

CC 38 (Heterocyclic Compounds (More Than One Hetero Atom))

IT Oxidation
Sulfonation
(of 1,2,5-selenadiazoles and 1,2,5-thiadiazoles)

IT Amines
(reactions of di-, with N-sulfinylaniline and thionyl chloride)

IT Barbituric acid, [[[5-(o-chlorophenyl)-1,3,4-thiadazol-2-yl]amino]methyl]thio-di-m-tolyl-
Barbituric acid, [[[5-(p-methoxyphenyl)-1,3,4-thiadiazol-2-yl]amino]methyl]thio-di-m-tolyl-

IT 288-39-1, 1,2,5-Thiadiazole 288-40-4, 1,2,5-Selenadiazole
(oxidation and sulfonation of)

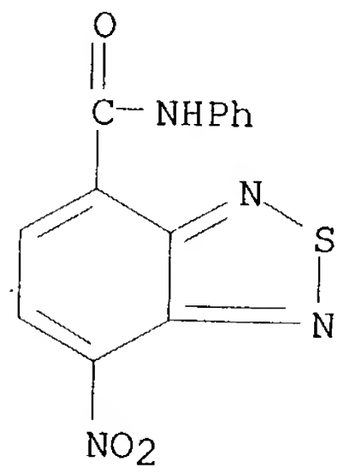
IT 2255-80-3, 2,1,3-Benzothiadiazole, 4-bromo-7-methyl- 3529-17-7, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-amino-, ethyl ester **3529-18-8**, 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro- 3529-30-4, 2,1,3-Benzothiadiazole-4-sulfonic acid, 7-methyl-, methyl ester 3529-31-5, 2,1,3-Benzothiadiazole-4-sulfonic acid, 7-methyl-, propyl ester 3529-32-6, 2,1,3-Benzothiadiazole-4-sulfonic acid, 5-methyl-, methyl ester 3529-33-7, 2,1,3-Benzothiadiazole-4-sulfonanilide, 7-methyl- 3529-34-8, 2,1,3-Benzothiadiazole-4-sulfonamide, 7-methyl-N-(α-methylphenethyl)- 3529-35-9, 2,1,3-Benzothiadiazole-4-sulfonamide, 5-methyl- 3529-36-0, 2,1,3-Benzothiadiazole-4-sulfonamide, N,5-dimethyl- 3529-37-1, 2,1,3-Benzothiadiazole-4-sulfonanilide, 5-methyl- 3529-38-2, 2,1,3-Benzothiadiazole-4-sulfonamide, 5-methyl-N-(α-methylphenethyl)- **3529-40-6**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, 2-(diethylamino)ethyl ester, hydrochloride 3529-53-1, 2,1,3-Benzothiadiazole-4-sulfonic acid, 5-methyl- 3529-55-3, 2,1,3-Benzothiadiazole-4-sulfonyl chloride, 7-methyl- 3529-56-4, 2,1,3-Benzothiadiazole-4-sulfinic acid, 7-methyl- 3529-57-5, 2,1,3-Benzothiadiazole-4-carboxylic acid **3529-58-6**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro- 3529-59-7, 2,1,3-Benzothiadiazole-5-carboxylic acid, 4-nitro- 3529-60-0, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-bromo- **3529-61-1**, 2,1,3-Benzothiadiazole-4-carbonyl chloride, 7-nitro- **3529-71-3**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, methyl ester **3529-72-4**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, ethyl ester **3529-73-5**, 2,1,3-Benzothiadiazole-4-carboxylic acid,

7-nitro-, propyl ester **3529-74-6**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, butyl ester 3660-43-3, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-chloro- 3662-82-6, 2,1,3-Benzothiadiazole-4-sulfonyl chloride, 5-methyl- 3663-14-7, 2,1,3-Benzothiadiazole-4-sulfonic acid, 5-methyl-, ethyl ester 3663-15-8, 2,1,3-Benzothiadiazole-4-sulfonamide, 7-methyl- **3663-16-9**, 2,1,3-Benzothiadiazole-4-carboxamide, N-(α-methylphenethyl)-7-nitro- 3746-14-3, 2,1,3-Benzothiadiazole-4-sulfonamide, N,7-dimethyl- 4061-69-2, 2,1,3-Benzothiadiazole-4-sulfonic acid, 5-methyl-, sodium salt 4752-27-6, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-amino-, 2-(diethylamino)ethyl ester, hydrochloride **31097-02-6**, 2,1,3-Benzothiadiazole-4-carboxyphenetidide, 7-nitro-
(preparation of)

IT **3529-18-8**, 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro-
3529-40-6, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, 2-(diethylamino)ethyl ester, hydrochloride **3529-58-6**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro- **3529-61-1**, 2,1,3-Benzothiadiazole-4-carbonyl chloride, 7-nitro- **3529-71-3**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, methyl ester **3529-72-4**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, ethyl ester **3529-73-5**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, propyl ester **3529-74-6**, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, butyl ester **3663-16-9**, 2,1,3-Benzothiadiazole-4-carboxamide, N-(α-methylphenethyl)-7-nitro- **31097-02-6**, 2,1,3-Benzothiadiazole-4-carboxyphenetidide, 7-nitro-
(preparation of)

RN 3529-18-8 HCAPLUS

CN 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro- (7CI, 8CI) (CA INDEX NAME)



RN 3529-40-6 HCAPLUS

CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, 2-(diethylamino)ethyl ester, hydrochloride (7CI, 8CI) (CA INDEX NAME)

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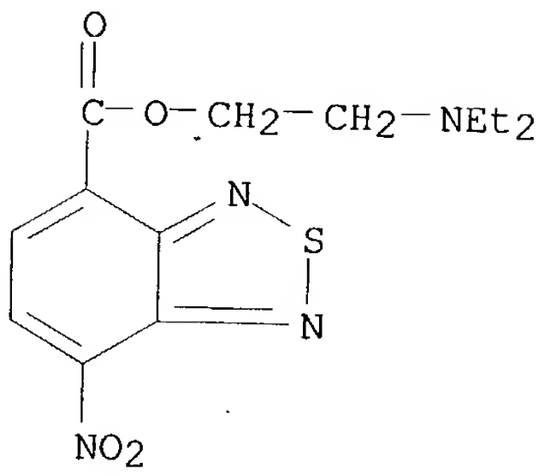
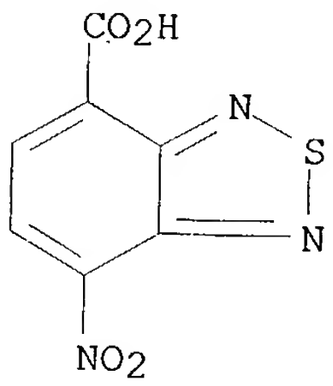
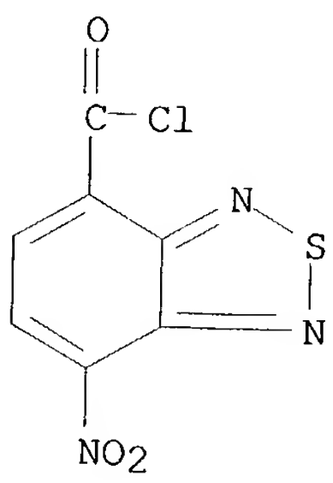


● HCl

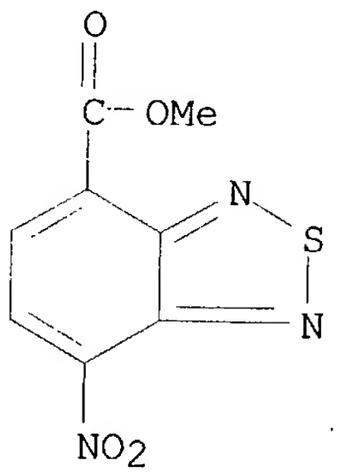
RN 3529-58-6 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro- (7CI, 8CI) (CA INDEX NAME)



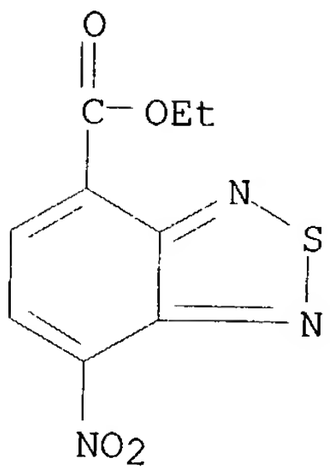
RN 3529-61-1 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carbonyl chloride, 7-nitro- (7CI, 8CI) (CA INDEX NAME)



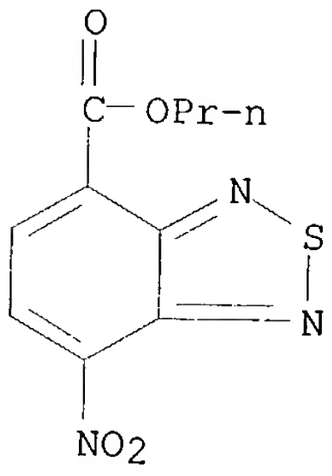
RN 3529-71-3 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, methyl ester (7CI, 8CI) (CA INDEX NAME)



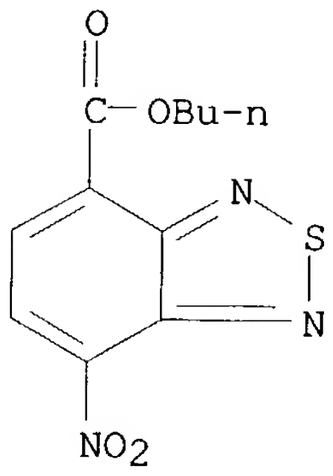
RN 3529-72-4 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, ethyl ester (7CI, 8CI) (CA INDEX NAME)



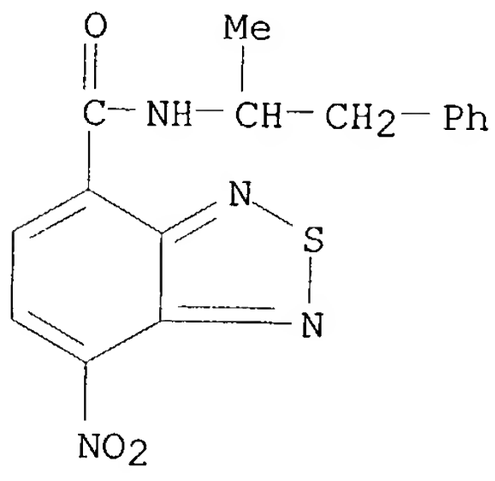
RN 3529-73-5 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, propyl ester (7CI, 8CI) (CA INDEX NAME)



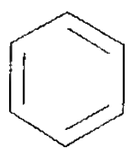
RN 3529-74-6 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, butyl ester (7CI, 8CI) (CA INDEX NAME)

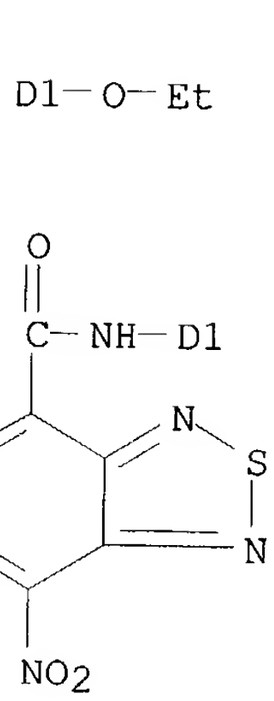


RN 3663-16-9 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxamide, N-(α-methylphenethyl)-7-nitro- (7CI, 8CI) (CA INDEX NAME)



RN 31097-02-6 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxanilide, ethoxy-7-nitro- (8CI) (CA INDEX NAME)





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L42 ANSWER 12 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1964:411331 HCAPLUS
DN 61:11331
OREF 61:1853b-d
TI Chemistry of 2.1.3-Thia- and selenadiazoles. XXIX. Synthesis and properties of 4- and 5-bromomenthyl-2,1,3-benzothiadiazoles
AU Pesin, V. G.; Vitenberg, I. G.; Khaletskii, A. M.
SO Zhurnal Obshchei Khimii (1964), 34(4), 1272-6
CODEN: ZOKHA4; ISSN: 0044-460X
DT Journal
LA Unavailable
AB 4-Methyl-2,1,3-benzothiadiazole and N-bromosuccinimide in CCl4 12 hrs. at reflux gave 70% 4-bromomethyl-2,1,3-benzothiadiazole (I), m. 90.5-1.5°; the yield was 55% in the presence of Bz2O2 in 1.5 hrs. The product was a lacrimator. Heated 3 hrs. with aqueous alc. KCN I gave the 4-cyanomethyl analog, m. 191°, which refluxed 4 hrs. with aqueous AcOH-H2SO4 gave the 4-carboxymethyl analog (II), m. 129-30°; the anilide m. 135.5-6.5°. Nitration of I with fuming HNO3 1 hr. at 0° gave 100% the 7-nitro derivative, m. 119-20°, also formed by bromination, as above, of the 7-nitro-4-methyl analog. The cyanomethyl derivative and HNO3 as above gave 100% the 7-nitro analog (III), m. 145-6°, also formed from the bromomethyl analog and KCN in 2 hrs. III was hydrolyzed with AcOH-concentrated HCl in 4 hrs. to 4-carboxymethyl-7-nitro-2,1,3-benzothiadiazole, m. 155°, also formed by nitration of II with fuming HNO3 1 hr. at room temperature; the anilide m. 178° (decomposition). Heating I with aqueous K2CO3 gave 4-hydroxymethyl-2,1,3-benzothiadiazole, m. 66-7°; alc. KOH similarly gave the 4-ethoxymethyl analog, m. 55-6°, while KCNS in aqueous Me2CO gave in 3 hrs. 4-thiocyanatomethyl-2,1,3-benzothiadiazole, m. 51°. 5-Bromomethyl-2,1,3-benzothiadiazole nitrated with HNO3 (d. 1.36) and concentrated H2SO4 1 hr. at 0-2° gave 58% the 4-nitro derivative, m. 126-7°, also formed by bromination of the 4-nitro-5-methyl analog. 5-Carboxymethyl-1,2,3-benzothiadiazole m. 159-60°; the anilide m. 183°. Similarly were prepared 5-hydroxymethyl-2,1,3-benzothiadiazole, m. 53-4°, the 5-ethoxymethyl analog, n20D 1.5949, the 5-cyanomethyl analog, m. 53-4°, the 5-ethoxymethyl analog, n20D 1.5949, the 5-cyanomethyl analog, m. 158-9°, and the 5-thiocyanatomethyl analog, m. 114-15°.
CC 38 (Heterocyclic Compounds (More Than One Hetero Atom))
IT Lacrimators
(4-(bromomethyl)-2,1,3-benzothiadiazole as)
IT 16405-99-5, 2,1,3-Benzothiadiazole, 4-(bromomethyl)- 16406-00-1, 2,1,3-Benzothiadiazole-4-methanol 16406-01-2, 2,1,3-Benzothiadiazole-4-acetonitrile 16406-02-3, Thiocyanic acid, 2,1,3-benzothiadiazol-4-ylmethyl ester 16406-02-3, 2,1,3-Benzothiadiazole, 4-(thiocyanatomethyl)- 42816-77-3, 2,1,3-Benzothiadiazole-4-acetic acid 55937-37-6, 2,1,3-Benzothiadiazole-5-acetic acid 65858-50-6, 2,1,3-Benzothiadiazole, 5-(bromomethyl)- 89488-04-0, 2,1,3-Benzothiadiazole-4-sulfonamide **89583-77-7**, 2,1,3-Benzothiadiazole, 4-(bromomethyl)-7-nitro- 89583-78-8, 2,1,3-Benzothiadiazole, 5-(bromomethyl)-4-nitro- 89795-51-7, 2,1,3-Benzothiadiazole-5-methanol **89899-01-4**, 2,1,3-Benzothiadiazole-4-acetic acid, 7-nitro- 89899-11-6, 2,1,3-Benzothiadiazole-5-acetonitrile 90557-43-0, 2,1,3-Benzothiadiazole, 4-(ethoxymethyl)- 90557-44-1, 2,1,3-Benzothiadiazole, 5-(ethoxymethyl)- **92061-28-4**, 2,1,3-Benzothiadiazole-4-acetanilide, 7-nitro- 92164-36-8, 2,1,3-Benzothiadiazole-4-acetanilide 92164-37-9, 2,1,3-Benzothiadiazole-5-acetanilide 93049-53-7,

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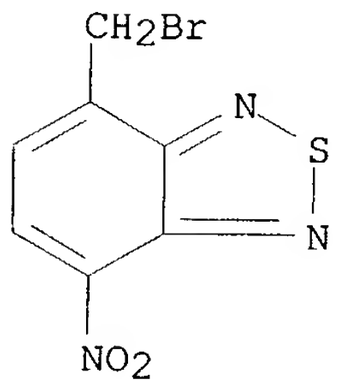
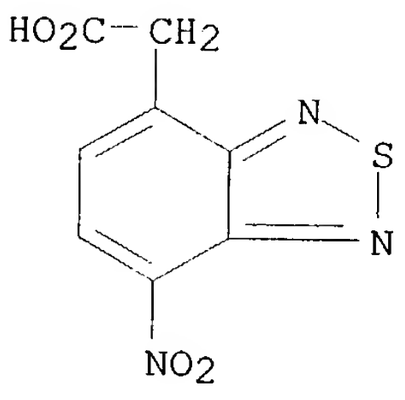
2,1,3-Benzothiadiazole, 5-(thiocyanatomethyl)- 93049-53-7, Thiocyanic acid, 2,1,3-benzothiadiazol-5-ylmethyl ester **93408-86-7**, 2,1,3-Benzothiadiazole-4-acetonitrile, 7-nitro-
(preparation of)

IT **89583-77-7**, 2,1,3-Benzothiadiazole, 4-(bromomethyl)-7-nitro-
89899-01-4, 2,1,3-Benzothiadiazole-4-acetic acid, 7-nitro-
92061-28-4, 2,1,3-Benzothiadiazole-4-acetanilide, 7-nitro-
93408-86-7, 2,1,3-Benzothiadiazole-4-acetonitrile, 7-nitro-
(preparation of)

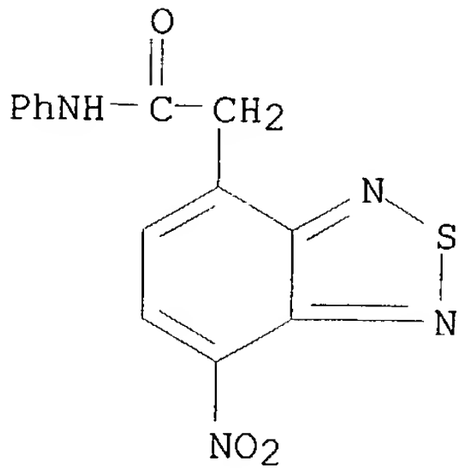
RN 89583-77-7 HCAPLUS
CN 2,1,3-Benzothiadiazole, 4-(bromomethyl)-7-nitro- (7CI) (CA INDEX NAME)



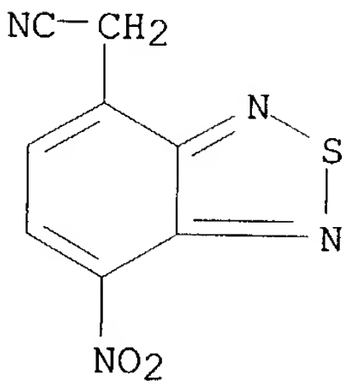
RN 89899-01-4 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-acetic acid, 7-nitro- (7CI) (CA INDEX NAME)



RN 92061-28-4 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-acetanilide, 7-nitro- (7CI) (CA INDEX NAME)



RN 93408-86-7 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-acetonitrile, 7-nitro- (7CI) (CA INDEX NAME)



L42 ANSWER 13 OF 13 HCAPLUS COPYRIGHT 2004 ACS on STN
AN 1964:60873 HCAPLUS
DN 60:60873
OREF 60:10671f-h,10672a-c
TI Benzo-2,1,3-thiadiazolecarboxylic acids
AU Muravnik, R. S.
SO Trudy Leningradskogo Khimiko-Farmatsevticheskogo Instituta (1962), (16), 184-93
CODEN: TLKFAD; ISSN: 0371-9235
DT Journal
LA Unavailable
GI For diagram(s), see printed CA Issue.
AB Benzo-2,1,3-thiadiazole-4(and 5)-carboxylic acids (I and II, resp.) and their derivs. were synthesized by the oxidation of 4- and 5-methylbenzo-2,1,3-thiadiazoles (III and IV, resp.) and corresponding derivs. Oxidation of the Me group of III and IV proceeds more successfully in the presence of electrophilic substituents in the nucleus, capable of weakening the influence of an adjacent hetero ring and strengthening the benzenoid properties of the carbon ring. Synthesized were the acid chloride, esters, and amides of 7-nitrobenzo-2,1,3-thiadiazole-4-carboxylic acid (V), from which were obtained the analogs of p-H2NC6H4CO2H with a thiadiazole ring, including analogs of Anesthesin and Novocaine. To a solution of 6 g. III in 50 ml. AcOH and 17 ml. H2SO4 (d. 1.84), 16 g. CrO3 was added (45-50°, 4 hrs.), the mixture on cooling poured into 150 ml. ice water, and the acid precipitated with 25 ml. 10% AgNO3; the 4.1 g. of

precipitate was treated with 20 ml. 10% HCl to give 0.8 g. unidentified material, C5H4N2O4S, m. 200-1° (H2O), and 2.8% I, m. 177.8-180°. To a solution of 6 g. 7-nitro-4-methylbenzo-2,1,3-thiadiazole in 75 ml. H2SO4 (d. 1.84) 13 g. K2Cr2O7 is added (42-45°, 2 hrs.) and the mixture stirred at 42-45°, 30 min. to give V, yield 75.5%, m. 237-8° (iso-PrOH). Oxidation of 4-nitro-5-methylbenzo-2,1,3-thiadiazole gave 4-nitrobenzo-2,1,3-thiadiazole-5-carboxylic acid (VI), yield 15.2%, m. 245-7° (decomposition) (alc.). To a solution of 2 g. 7-chloro-4-methylbenzo-2,1,3-thiadiazole in 50 ml. 98% AcOH and 7 ml. H2SO4 (d. 1.84), 3 g. CrO3 was added at 45-50° for 1 hr. and the mixture stirred at 45-50° 30 min. to give 7-chlorobenzo-2,1,3-thiadiazole-4-carboxylic acid, yield 38.8%, m. 254-5° (alc.). 7-Bromo-4-methylbenzo-2,1,3-thiadiazole oxidized with AcOH-H2SO4-CrO3 gave 51.8% 4-carboxy analog, m. 216-17° (70% AcOH). V (1 g.) in 10 ml. SOCl2 is heated to complete solution to give the acid chloride (VII), m. 94.5-6.0° (benzene). VII (0.3 g.) in 5 ml. absolute MeOH heated to complete solution gave V Me ester, m. 130.1-1.0° (1:3 AcOH-H2O). The Et ester of V m. 117-18°, the Pr ester of V, m. 87-8° (dilute AcOH), V anilide, m. 252-3° (98% AcOH), and V p-ethoxyanilide, m.

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202-3° (98% AcOH), were obtained. VI (1 g.) in 10 ml. SOCl2 was heated to complete solution excess SOCl2 removed, and the residue in absolute ether treated with PhNH2 to give VI anilide, 60%, m. 217-18° (70% AcOH). VII (1 g.) in 15 ml. CHCl3 and 0.6 g. β-phenylisopropylamine heated at 100° for 30 min. gave VII β-phenylisopropylamide, 59.3%, m. 126-7° (MeOH). A mixture of 40 ml. alc., 4 g. V Et ester, and 2.5 g. Fe filings was heated with stirring to 85°, gradually 50 ml. 1% AcOH added, and the mixture heated at 85° 30 min. to give Et ester of 7-aminobenzo-2,1,3-thiadiazole-6-carboxylic acid, C9H9N3O2S (VIII), yield 35.3%, m. 149-50° (H2O). To 12 g. VII in 10 ml. absolute C6H6, 6 g. Et2NCH2CH2OH (IX) was added rapidly with vigorous stirring to give V diethylaminoethyl ester (X), yield 87.5%, m. 184-5° (PrOH). At pH 8-9 X was hydrolyzed with formation of IX and V. To 9.2 g. in 40 ml. water, 40 ml. alc., 10 ml. AcOH, and 6 g. Fe filings were added and the mixture heated at 100° 30 min. the diethylaminoethyl ester of VIII.HCl, 24.8%, m. 214-15° (PrOH).

CC 38 (Heterocyclic Compounds (More Than One Hetero Atom))

IT 273-13-2, 2,1,3-Benzothiadiazole
(derivs.)

IT **3529-18-8**, 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro-
3529-57-5, 2,1,3-Benzothiadiazole-4-carboxylic acid **3529-58-6**,
2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro- 3529-59-7,
2,1,3-Benzothiadiazole-5-carboxylic acid, 4-nitro- 3529-60-0,
2,1,3-Benzothiadiazole-4-carboxylic acid, 7-bromo- **3529-61-1**,
2,1,3-Benzothiadiazole-4-carbonyl chloride, 7-nitro- **3529-71-3**,
2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, methyl ester
3529-72-4, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-,
ethyl ester **3529-73-5**, 2,1,3-Benzothiadiazole-4-carboxylic acid,
7-nitro-, propyl ester 3660-43-3, 2,1,3-Benzothiadiazole-4-carboxylic
acid, 7-chloro- **3663-16-9**, 2,1,3-Benzothiadiazole-4-carboxamide,
N-(α-methylphenethyl)-7-nitro- 16405-98-4, 2,1,3-Benzothiadiazole-
5-carboxylic acid 90349-26-1, 2,1,3-Benzothiadiazole-5-carboxylic acid,
4-amino-, ethyl ester 91805-05-9, 2,1,3-Benzothiadiazole-5-
carboxanilide, 4-nitro- 92034-89-4, 2,1,3-Benzothiadiazole-5-carboxylic
acid, 4-amino-, 2-(diethylamino)ethyl ester **92110-35-5**,
2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, 2-(diethylamino)ethyl
ester 95010-86-9, Benzo[1,2-d:4,5-d']diimidazole-2,4,6,8-tetrone,
1,3,5,7-tetrahydro-1,3-dimethyl- 95516-46-4, Benzo[1,2-d:4,5-
d']diimidazole-2,4,6,8-tetrone, 1,3,5,7-tetrahydro-1,3,5,7-tetramethyl-
97062-91-4, 2,1,3-Benzothiadiazole-4-carboxy-p-phenetidide, 7-nitro-
(preparation of)

IT **3529-18-8**, 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro-
3529-58-6, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-
3529-61-1, 2,1,3-Benzothiadiazole-4-carbonyl chloride, 7-nitro-
3529-71-3, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-,
methyl ester **3529-72-4**, 2,1,3-Benzothiadiazole-4-carboxylic
acid, 7-nitro-, ethyl ester **3529-73-5**, 2,1,3-Benzothiadiazole-4-
carboxylic acid, 7-nitro-, propyl ester **3663-16-9**,
2,1,3-Benzothiadiazole-4-carboxamide, N-(α-methylphenethyl)-7-nitro-
92110-35-5, 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-,
2-(diethylamino)ethyl ester
(preparation of)

RN 3529-18-8 HCAPLUS

CN 2,1,3-Benzothiadiazole-4-carboxanilide, 7-nitro- (7CI, 8CI) (CA INDEX NAME)

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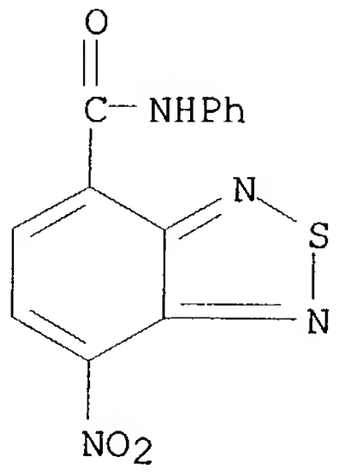
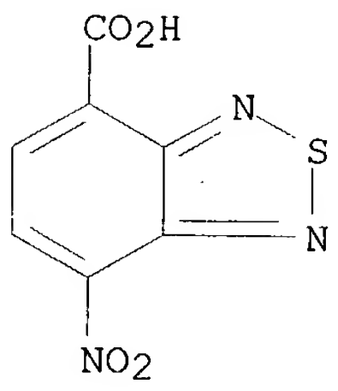
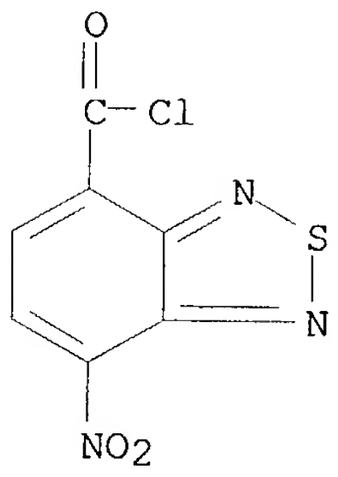


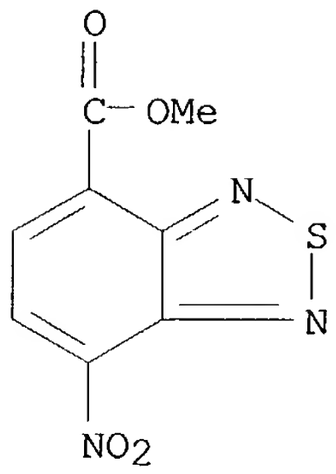
RN 3529-58-6 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro- (7CI, 8CI) (CA INDEX NAME)



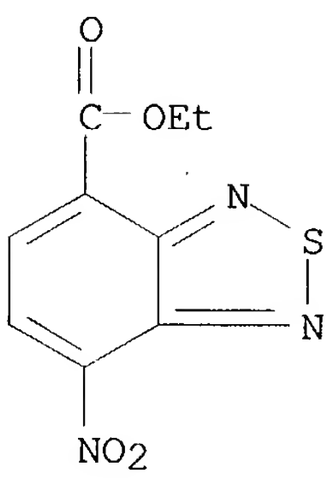
RN 3529-61-1 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carbonyl chloride, 7-nitro- (7CI, 8CI) (CA INDEX NAME)



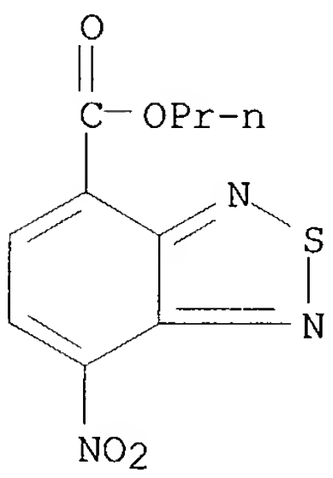
RN 3529-71-3 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, methyl ester (7CI, 8CI) (CA INDEX NAME)



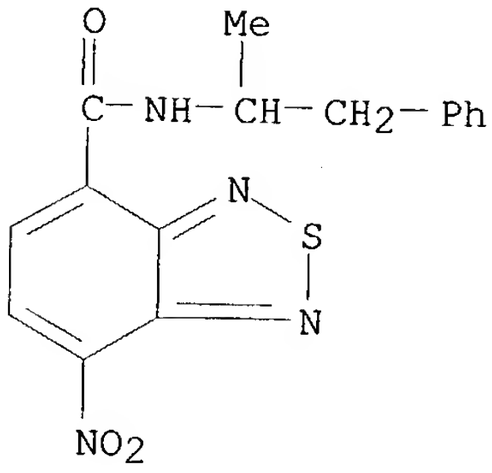
RN 3529-72-4 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, ethyl ester (7CI, 8CI) (CA INDEX NAME)



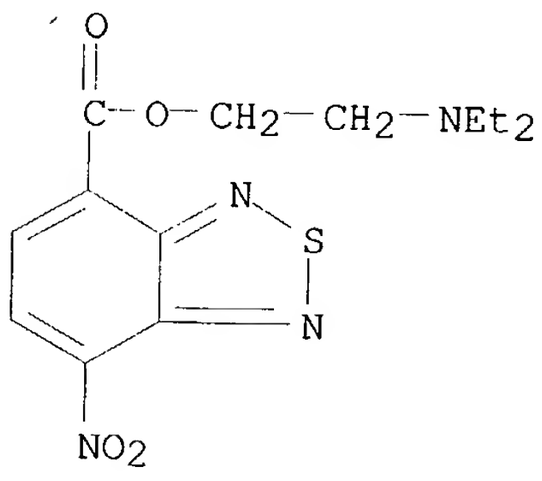
RN 3529-73-5 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, propyl ester (7CI, 8CI) (CA INDEX NAME)



RN 3663-16-9 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxamide, N-(α-methylphenethyl)-7-nitro- (7CI, 8CI) (CA INDEX NAME)



RN 92110-35-5 HCAPLUS
CN 2,1,3-Benzothiadiazole-4-carboxylic acid, 7-nitro-, 2-(diethylamino)ethyl ester (7CI) (CA INDEX NAME)



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